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Doctor's Dissertation

A Study of the Viscoelastic Properties
of Paper by means of
Tensile Creep Tests

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A STUDY OF THE VISCOELASTIC PROPERTIES OF PAPER
BY MEANS OF TENSILE CREEP TESTS

A thesis submitted by

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TABLE OF CONTENTS

	Page
PRESENTATION OF THE PROBLEM	1
INTRODUCTION AND HISTORICAL REVIEW	5
Prerupture Response to Stress	6
Immediate Elastic Deformation	7
Delayed Elastic Deformation	9
Configurational Elastic Response	10
Reversible Crystallization	12
Nonrecoverable Deformation	13
Viscous Flow	13
Irreversible Crystallization	14
Metastability	15
Mechanical Tests	16
The Creep Test	17
Analysis of Creep Data	18
Mathematical Equations and the Creep Function	18
Mechanical Analogies	19
Boltzmann's Superposition Principle	19
The Relation Between Mechanical Behavior and Structure	21
The Viscoelastic Properties of Paper	26
APPARATUS	34
Creep Testing Apparatus	34
Apparatus for Control of Relative Humidity	37
GENERAL EXPERIMENTAL PROCEDURES	39
Preparation of Specimens	39
Characterization of Handsheets	41

Creep Testing Procedures	46
FIRST-CREEP PROPERTIES OF ALPHA PULP HANDSHEETS	49
First Creep	50
The Master Creep Curve	56
The Timeshift Requirement in the Construction of the Master Creep Curve	60
Total First-Creep Deformation Versus Initial Stress at Constant Times	64
First-Creep Mechanisms of Response	64
THE RELATIONSHIP BETWEEN CREEP AND LOAD-DEFORMATION TESTS	67
CREEP RECOVERY PROPERTIES OF ALPHA PULP HANDSHEETS	79
Creep Recovery as a Function of Residual Load	80
Creep Recovery <u>Versus</u> Duration of First-Creep Test	83
Creep Recovery <u>Versus</u> Initial Stress	87
MECHANICAL CONDITIONING AND PRIMARY CREEP	92
Second-Creep Response as a Function of the First-Creep Test	93
Long-Duration Multiple-Cycle Tests	98
Typical Multiple-Cycle Test at 50% R.H.	99
Primary Creep as a Function of Stress	108
DEPENDENCE OF CREEP PROPERTIES ON SHEET STRUCTURE	116
Experimental Procedures	117
Results and Discussion	118
First-Creep Properties	118
First-Recovery Properties	128
CREEP PROPERTIES <u>VERSUS</u> RELATIVE HUMIDITY	132
Experimental Procedures	133

Results and Discussion	135
Apparent Modulus of Elasticity	136
First-Creep Response	136
Master Creep Curves	136
Deformation-Time-Initial Stress Relationships	146
First-Recovery Response	152
THE EFFECT OF HUMIDIFICATION ON CREEP PROPERTIES	158
SWELLING RECOVERY	171
HYGROEXPANSIVITY	177
SUMMARY AND CONCLUSIONS	183
LITERATURE CITED	195
APPENDIX	199

PRESENTATION OF THE PROBLEM

In the manufacture of paper, one has some control of the mechanical properties and structural characteristics of the finished product. The problem of relating processing variables and paper properties is important from both a practical and a research viewpoint. A vast amount of experimental work in this general area has provided the broad empirical relationships, but has contributed less toward an understanding of the mechanisms involved. To a large extent, this can be traced to the lack of a precise description of the structure of paper.

For many years, the mechanical properties of paper have been defined primarily by rupture tests. It was generally recognized that the rate of testing influenced the results and attempts were made to control the rate of stress or strain development prior to rupture. More recently, increased attention has been directed toward investigation of the prerule response to stress, chiefly by means of the load-deformation test. Prerule mechanical properties have been employed in evaluating variables of all types in the manufacture of pulp and paper. Interpretation of the results, however, was often limited by the lack of a sound basis for relating prerule mechanical properties to the structural characteristics of the sheet. It is the purpose of this study to develop information which will lead to a better understanding of those relationships.

It is generally recognized that the mechanical properties of paper are functions of both the sheet structure and the properties of the component fibrous raw materials. In paper, as in all materials, the response to an external stress should be relatable, ultimately, to the

molecular structure and the shape of the solid specimen. This suggests the use of stress response as a means of elucidating molecular structure and structural changes. Unfortunately, the complex and heterogeneous void pattern of paper precludes the specification of a simple shape factor. Direct determination of the mechanical properties of papermaking fibers is extremely difficult experimentally, and little is known about the relationships between stress response and the molecular structure of highly oriented and partially crystalline native cellulosic polymers. The possibility of relating structure to stress response, therefore, is largely academic since one cannot define either the sheet structure or the fiber properties by independent methods. Nevertheless, the dependency of the response to stress on the shape and molecular structure of the specimen forms the basic precept on which an approach to the overall problem should be based.

Before any attempts can be made to relate the structure of paper to its mechanical behavior, it is necessary to develop the fundamental relationships between time, deformation, and stress, and to define the individual mechanisms of response to stress. The tensile creep test at constant load in the plane of the sheet has been selected as a means of providing this information. The tensile creep test cannot completely describe the mechanical properties of a material as structurally heterogeneous as paper; however, it meets the important requirement of separating the variables of time, deformation, and stress.

Mechanically, paper is classified as a viscoelastic material. Its response to stress is characterized by an immediate elastic deformation

and a series of delayed deformations, which are distributed in time and only partly elastic. The creep properties which will be investigated include the first-creep response, the first-recovery response and the response in subsequent tests. Following the preliminary work, creep properties will be determined for handsheets of different solid fraction produced by varying degrees of beating and wet pressing. This study will permit an assessment of the earlier results in terms of their general applicability to papers which vary primarily in sheet structure.

Paper, as normally tested and used, is a water-plasticized polymer. Its mechanical properties are strong functions of its moisture content. It is unlikely that an understanding of the mechanical behavior and structure of paper could be obtained without an understanding of the effect of moisture content on mechanical behavior, hence, creep properties will be determined over a range of relative humidities. These results should aid in separating and possibly defining the different mechanisms by which paper deforms under tensile loads.

TABLE I

NOMENCLATURE

(summary of the more important symbols)

\underline{a}	exponent of time in exponential equations describing early first-creep response, dimensionless
\underline{A}_0	initial solid cross-sectional area of specimen at 50% R.H., and 73°F. calculated from assumed fiber density of 1.5 g./cc., sq. mm.
\underline{E}_a	apparent modulus of elasticity for tensile loads in the plane of the sheet, kg./sq. mm.
\underline{K}	constant of proportionality in logarithmic equation describing first-creep response, dimensionless
\underline{L}_0	initial specimen length, inches
\underline{P}	load of test, kg.
\underline{S}_0	calculated average initial stress ($\underline{P}/\underline{A}_0$), kg./sq. mm.
\underline{t}	time of test measured from instant of application or removal of load, sec.
\underline{y}	total deformation in test, inches
\underline{y}_0	immediate elastic deformation in test, inches
\underline{y}_c	total delayed deformation in creep test, inches

INTRODUCTION AND HISTORICAL REVIEW

Studies of the mechanical properties of solids are extremely broad and varied in the literature. The bulk of the earlier investigations deal with structural materials and properties important to structural applications, chiefly, ultimate strength and the various moduli of elasticity. In more recent years, following the rapid application and development of synthetic textile filaments, increased attention has been directed toward prerule mechanical tests. Even more recent are the attempts to correlate mechanical properties with molecular and macroscopic structure, the influence of processing conditions, and with external variables such as temperature and relative humidity.

For paper, studies of prerule response to stress are not numerous and have occurred largely within the last decade. Consequently, one must draw on the relationships developed for materials similar in molecular structure. Paper is unique in over-all structure, hence, analogies with other polymeric materials must be drawn with care. One becomes interested, however, in materials of partially crystalline molecular structure characterized by a relatively high degree of intermolecular bonding. These include native fibers such as cotton and ramie, the cellulose derivatives such as cellulose acetate and viscose rayon, and synthetic textile filaments such as nylon.

The purpose of this review is first to familiarize the reader with the general principles of response to stress with emphasis on the creep test. Secondly, the relation between prerule response and structure is discussed, and finally, the past work relating to the viscoelastic properties of paper is reviewed.

PRERUPTURE RESPONSE TO STRESS

A solid subjected to an external load will respond by a deformation in a manner tending to relieve the load. The deformation will be determined by the magnitude of the applied load, its manner of application, the molecular structure and the true shape of the specimen. Shape is delineated by the surface outline of the solid material and not necessarily by the gross apparent dimensions of the test specimen. All factors other than the molecular structure and composition of the specimen must be measurable if it is desired to determine the mechanical properties of the substance. In some instances, it is not possible to provide for uniformity in the true shape of the solid specimen or in the applied load, and the mechanical properties must be determined for the over-all structure considered as an entity. Essentially, that is the case for paper and other materials of porous, heterogeneous macroscopic structure.

The fundamental molecular mechanisms to which the observed deformation can be attributed provides the key to relating mechanical properties and structure. Time-dependent deformations are the result of mechanisms involving structural changes. The rate of structural change determines the observed rate of deformation. As deformation occurs, the structure of the specimen changes with an attendant change in mechanical properties. Several mechanisms of response may occur simultaneously and may be interdependent. Mechanical tests alone, therefore, may not furnish sufficient information to establish the various mechanisms of response.

The total deformation at any time following the application of a load may be divided into three general types of response. These include the

immediate elastic deformation, the delayed elastic deformation, and the nonrecoverable deformation. Immediate elastic deformation is the perfectly elastic deformation which is considered to occur instantaneously with the application of load. It is not time dependent and is considered to be recovered instantly upon removal of the load. Delayed elastic deformation is that deformation which occurs distributed in time after the application of load, and which is recoverable (also distributed in time) within reasonable periods of time after removal of the load. Nonrecoverable deformation is defined as that portion of the total deformation which is not recoverable within reasonable periods of time at the test conditions following removal of the load. Nonrecoverable deformation may be classified further into groups according to its relative permanence. All three general types of response are common to most organic high polymers. The following discussion relates to the various types of deformation common to polymeric materials with emphasis on the mechanisms of response.

IMMEDIATE ELASTIC DEFORMATION

The immediate elastic deformation is the result of the deformation of primary valence bonds, the changes in primary valence bond angles, and the extensions of the various secondary bonds. Upon application of load, this very rapid elastic deformation is considered to occur instantaneously.

The modulus of elasticity is defined as the ratio of the applied stress to the resulting immediate elastic strain. It must be further defined by the type of stress which is applied and the nature of the strain which is measured. It is frequently a very difficult quantity to measure accurately in the presence of extremely short-time delayed deformations. The value of the elastic modulus is determined by the relative contributions of the

various mechanisms of deformation. Since it is much easier to deform secondary bonds, the modulus of elasticity will be lowest for materials in which the numbers of these bonds per unit of volume is greatest. Meyer and Lotmar (1) have calculated the modulus of elasticity in tension for crystalline cellulose in the order of 15×10^6 p.s.i. (about 10,000 kg./sq. mm.) Mark (2) has compiled a summary of the elastic moduli of various polymers in tension. Except for the highly oriented and highly crystalline native fibers such as ramie, hemp, and flax, the elastic modulus of cellulose and cellulose derivatives is much lower than the calculated value for crystalline cellulose, and may range from 100 to 5000 kg./sq. mm. for various regenerated cellulose fibers. Leaderman (3) determined the modulus of elasticity of cellulose acetate to be 576 kg./sq. mm., unstretched viscose rayon to be 1050 to 1094 kg./sq. mm., and wet stretched and re-dried viscose rayon to be 1420 kg./sq. mm. Wakeham and Honald (4) reported elastic moduli for cotton fibers ranging from 740 to 1360 kg./sq. mm. Values are not available for individual wood pulp fibers.

The elastic modulus of paper in tension has not been well defined in the literature, since attention has been directed largely to other mechanical properties. Steenberg (5) reported an apparent elastic modulus of 100 to 200 kg./sq. mm. for cross machine-direction specimens of paper prepared from sulfite pulp based on the measured thickness of the specimen. These values would probably be twice as large if based on the solid cross-sectional area of the specimen. The apparent elastic modulus may be considerably greater in the machine direction. Keeney (6) determined the initial slopes of load-deformation curves for handsheets prepared from semichemical pulp at various degrees of beating and wet pressing. Apparent

elastic moduli calculated from his data range between 700 and 1000 kg./sq. mm.

The elastic modulus of paper was reduced by the addition of urea and substituted-urea compounds to paper sheets as indicated by Fisher (7). Andersson and Berkyto (8) demonstrated that large decreases in elastic modulus occur with increasing moisture content of paper. Increases in elastic modulus were reported by Ivarsson (9) following mechanical conditioning of paper specimens.

A lower elastic modulus is related to higher percentages of amorphous material and greater heterogeneities in the structure of polymers. There appears to be a general inverse relationship between the elastic modulus and the delayed deformation of polymers. The relationship is too vague and unpredictable to be of much value in relating creep properties to structure. It is generally conceded that the immediate elastic deformation is difficult to interpret in terms of the various mechanisms to which it is attributed.

DELAYED ELASTIC DEFORMATION

In organic high polymers, the delayed elastic deformation may comprise a large portion of the total deformation. All of the delayed deformation which is recoverable at the test conditions of temperature and relative humidity following the removal of load will be termed delayed elastic deformation. Thus, the condition of recoverability is used as one of the criteria for separating the various types of response to stress. Delayed elastic deformation is due principally to configurational elastic response.

Reversible changes of phase, however, may also provide for recoverable deformations. The following very brief review of the configurational and other mechanisms of response is taken from Alfrey (10).

Configurational Elastic Response

The packing of molecules in the amorphous regions of high polymers is less than the maximum, hence, the solid is considered to have holes of molecular dimensions throughout the amorphous regions. The molecules in these regions are kinked, twisted, partially-coiled, etc., in contrast to the orderly aligned arrangement of the crystalline regions. Each molecule is bonded to other molecules at many points along its length, but because of the random orientation, molecular segments of varying length will be relatively unbonded. Molecular segments adjacent to "holes" may jump to new positions when they gain sufficient energy to overcome the forces which previously delineated their vibrational paths. Such movements of molecular segments may require the rupture of secondary valence bonds followed by the formation of new bonds in new positions. If a polymer is in a state of configurational equilibrium, the segmental jumps are random in direction and no change in specimen shape will occur with time. Under the influence of an external stress, however, segmental jumps will be more frequent in a direction tending to relieve the applied stress, since less energy will be required to make those jumps. This effect is described in terms of an unsymmetrically biased molecular force field which results in a decreased activation energy for segmental movement in the direction of the stress. The accumulated, biased segmental jumps under the action of an external stress result in a more orderly molecular

configuration. The molecular chains are partially uncoiled, unkinked, and generally untangled. Upon removal of the external stress, the molecules return slowly to the more random configuration, since the more random configurational positions are statistically predominant. This is manifested as a time-dependent contraction in length of the specimen. In simplest form, changes in molecular configuration are constant volume processes, and are characterized thermodynamically solely as entropy changes.

An amorphous polymer which responds to stress in a manner consistent with a Hookian spring and Newtonian viscosity is described as a linear polymer. Partially crystalline polymers are nonlinear; the mechanisms of configurational response are fundamentally the same, but are complicated by the restraints offered by crystalline portions of the polymer.

The ratio of the viscosity to the elastic modulus is designated as the relaxation time or retardation time depending on the mechanical test and mechanical analog which is considered. Retardation time is applied to creep at constant stress, which is represented phenomenologically by an elastic spring and viscous dashpot in parallel (Voight element). Relaxation time is applied to the decay of stress with time and is represented by a spring and dashpot in series (Maxwell element). Relaxation and retardation times are mathematically identical. Different retardation times arise because of differences in the size and character of molecular segments. The configurational elastic response of many polymers is described best by several characteristic retardation times or by a continuous distribution. The distribution curve may be approximated by a plot of the configurational elastic response that occurs in successive increments

of time versus the average time of each increment. Curves of this type exhibit maximums if the deformation-log time curve is sigmoidal.

A theory relating the delayed elastic response of textile fibers to the dimensions of a molecular flow unit and a specific rate constant related to the free energy of activation for viscous flow has been developed by Halsey, White, and Eyring (11), and discussed later by Halsey (12). A three-element mechanical model consisting of a Maxwell unit in parallel with an elastic element was found to describe the response of many textiles. Non-Newtonian dashpot fluids are required.

Reversible Crystallization

Leaderman (3) has suggested that reversible crystallite growth may be a mechanism of response in the delayed elastic deformation of nylon at high stresses. He postulates that the straightening of the molecular chains reaches a point where crystallite growth at the expense of amorphous material becomes possible. The nylon filament was "stiffer" for removal of load than during its application. This effect was noted by recovery curves which were lower in total deformation at early times and steeper in slope than the preceding creep curves. Leaderman attributed this behavior to reductions in the percentage of amorphous material during the creep test because of crystallization, which reduced the amount of configurational elastic recovery at early times. The stress-induced crystalline structure melted during the recovery period and the creep deformation was entirely recoverable at a 1:1 ratio of recovery time to creep time.

Evidence for crystallization was indirect and based on the departure from the characteristic creep and recovery behavior noted at lower loads.

In addition to the distinctly steeper recovery curve compared to the creep curve, a simple upward displacement of the creep curves of nylon along the deformation axis occurred with increasing load at higher loads. This independence of the delayed response on load was also attributed to crystallization. Leaderman reported similar behavior for viscose rayon at high relative humidities. Effects of this type are also indicated by the creep data of viscose rayon reported by Press (13), and acetate rayon by Mark and Press (14).

NONRECOVERABLE DEFORMATION

Nonrecoverable deformation of polymers is defined as that portion of the total deformation which is not recoverable at the test conditions of temperature and relative humidity following removal of the applied load. The nonrecoverable deformation may be characterized partly in terms of the relative permanence of these deformations. If chemical changes during the test period are eliminated from consideration, truly permanent deformations are limited to viscous flow and irreversible crystallization. A portion of the configurational elastic response of crystalline polymers or of the additional crystallization might be nonrecoverable at the test conditions but recoverable if the polymer is swollen by raising the moisture content or by increasing the temperature. Deformations which are ultimately recoverable by swelling treatments cannot be due to viscose flow.

Viscous Flow

The term viscous flow is applied rather indiscriminately to the response of polymeric materials. Customarily, the term is applied to truly

permanent deformations, which arise by actual transfers of entire molecules in amorphous polymers. Configurational elasticity was described as a mechanism of response involving jumps of molecular segments. Each segmental jump results in a slight change in the center of gravity of the entire molecule to which it belongs. Accumulated segmental jumps may give rise to a macro-Brownian movement of the entire molecule which may actually wander throughout the amorphous polymer. Deformation which is attributable to the transfer of molecules is true viscous flow and is not recoverable. It is generally agreed that deformation of this type is not possible for the typical crystalline polymer. Molecules are so firmly bonded in the crystalline regions that relative movement of entire molecules is precluded by these juncture points. This is more plausible if viewed in the light of the fringe theory of structure. The molecules are much longer than the length of a crystallite and are considered to pass through many regions of crystalline and amorphous structure. True viscous flow, therefore, is limited to polymers in which entire molecules exist in an amorphous continuum.

Irreversible Crystallization

Little is known about the mechanisms of crystallization in polymers other than rubber. The possibility of reversible crystallization has been discussed in terms of the data presented by Leaderman for nylon. Alfrey (10) suggested that there may be two types of crystallization. The growth of crystallites without change of polycrystalline structure may be reversible, whereas the additional crystallization accompanied by changes in polycrystalline structure may be irreversible. Mark (15) suggests that the nonrecoverable deformation of crystalline polymers may be due to crystal

growth in wet stretching and the fissuring of the crystals at their ends in dry stretching. Partial restoration of the original structure may then be possible by swelling of the polymer. Crystallization acts to aid the stress in extending the specimen and is unlikely to begin until the molecular chains have been straightened and aligned sufficiently to enable the intermolecular forces to complete the process.

Metastability

Polymers in which the intermolecular bonding forces per unit of molecular length are high, such as cellulose and the cellulose derivatives, are considered to be very susceptible to "frozen in" configurations. Any apparent equilibrium state in these polymers is likely to be a state of metastable equilibrium. Various combinations of crystallization and configurational changes may be nonrecoverable due to metastability. The new configurations are considered stable at the test conditions chiefly because of strong bonding forces which act to increase the resistance to recovery. The existing thermodynamic driving forces or residual stresses tending to promote recovery are too weak to result in a significant rate of recovery until the resistance is weakened by plasticization or by an increase of temperature.

MECHANICAL TESTS

The various methods of specifying the mechanical properties of viscoelastic materials have been reviewed by Alfrey and Doty (16). All describe the time-dependency of strain or stress. It is most desirable to specify mechanical properties in terms of the fundamental attributes of the material which may describe the response in a test of any type. More frequently, the mechanical properties are described by mapping out the response in a particular kind of test. This method of description is less satisfactory, but it must suffice if the mechanical behavior is complex.

The three most popular mechanical tests include the stress relaxation test, the creep test and the load-deformation test. Only the first two permit a separation of the variables of time, stress, and strain. The load-deformation test treats strain or stress in an arbitrary time-controlled sequence and the results of these tests are correspondingly more difficult to interpret.

In the stress-relaxation test, the rate of decay of stress is measured at constant deformation. A number of tests at varying levels of deformation may be required to delineate the stress-relaxation properties of a material. The constant-load creep test is the simplest mechanical test which can be applied to a polymer to provide a description of the time effects. It involves rapid application of a load to a specimen followed by measurement of deformation versus time. Creep tests over a range of loads are required. Attempts to relate the stress relaxation behavior to creep behavior mathematically have been made by Sips (17) for polyisobutylene.

It is an extremely difficult procedure and adequate chiefly if deformation is proportional to load. If the delayed deformation is large compared to the immediate elastic deformation, it will not be possible to relate the two tests in a simple manner.

THE CREEP TEST

An excellent historical survey of the creep test and the creep properties of high polymers was presented by Leaderman (3), with emphasis on reversible creep behavior. The following discussion is presented to familiarize the reader with the nomenclature and fundamental principles of creep testing.

A "creep curve" is a plot of deformation as a function of time. It is considered normally on a semilogarithmic plot with time measured from the instant of application of the load. For small deformations, constant load will approximate constant stress. The creep load is applied rapidly compared to the time of the first reading in order to minimize any possible effect due to the manner of load application. Creep data are obtained within a limited experimental time interval, ranging from the time of the first reading to the time of the last. Creep tests are generally discontinued before their logical conclusion, the cessation of creep deformation or rupture of the specimen.

"Total creep deformation" is the sum of the "immediate elastic deformation" and the "delayed deformation." The delayed deformation at any time cannot be calculated without knowledge of the immediate elastic deformation. This latter quantity can be estimated, in some cases, by extrapolating the creep curve to zero slope at early times. Frequently, it is necessary

to estimate this quantity from low-load static tests where the creep effects are small. When accurate measurements of the immediate elastic deformation cannot be made, it is useful to compare "relative delayed deformations" between two arbitrary times in the creep test.

Upon removal of the creep load at any time during the creep test, one measures the contraction in length in a "creep recovery" test. The "total recovery" includes both the immediate elastic contraction and the delayed recovery. The total delayed deformation in creep tests may be only partially recoverable. The recoverable component is termed "primary creep," and the nonrecoverable component is "secondary creep." It is often possible to study primary creep behavior by removing the secondary creep in one or more cycles of creep tests. Creep in subsequent tests may be almost totally recoverable. The process of subjecting a specimen to one or more cycles of creep and recovery to eliminate all or part of the secondary creep in subsequent tests is termed "mechanical conditioning." A specimen is said to be "mechanically conditioned" when the response of subsequent tests is largely recoverable. The specimen is normally considered to be mechanically conditioned, however, only for creep loads and test durations which are equal or less than those of the previous mechanical conditioning tests. Further secondary creep may occur if those limits are exceeded.

ANALYSIS OF CREEP DATA

Mathematical Equations and the Creep Function

It is often possible to describe all or part of the experimental creep curve by one or more mathematical equations. If the creep response is comprised of primary and secondary creep, several equations may be required to fit different parts of the creep curve. For primary creep, the deformation

versus log-time relationship will be represented by a sigmoidal curve which may be fitted by a single equation from zero deformation at zero time to some finite value of deformation at very long times. The function of time to which the delayed deformation is related is termed the "creep function." It is seldom possible to determine the creep function because of its complexity. Consequently, mathematical analysis has been limited to simple equations which apply to explicit parts of the creep curve.

Mechanical Analogies

Various combinations of elastic springs and viscous dashpots may be devised which will react under applied stress in a manner approximating the behavior of the specimen. If the response to stress is simple, the spring and dashpot analogy is very useful. In primary creep, the various parameters assigned to the springs and dashpots have the physical significance of the molecular segments to which the configurational response is attributed. As the creep curve becomes more complex, the model must become more complex and any physical significance between the model and polymer structure becomes vague. Little can be gained by mechanical models which empirically describe the mechanical behavior without relating the model to polymer structure. Mechanical models have been very popular in describing the primary creep behavior of linear amorphous polymers but are of less value in describing the more complex behavior shown by the crystalline polymers.

Boltzmann's Superposition Principle

The principle of superposition was originally proposed by Boltzmann (18) in 1876 to describe the mechanical behavior of various polymeric materials

in the absence of nonrecoverable deformations. The principle was extended to nonlinear primary creep response by Leaderman (3) and many of its implications were discussed. The superposition principle is an empirical description of mechanical behavior in which configurational elastic mechanisms of response are predominant. In effect, the principle states that in primary creep a substance will respond to a complicated sequence of loading and unloading as if each load were applied independently for an indefinite period of time, and that the deformation at any time during the test will be the simple summation of the deformations due to all of the loads in the sequence at that instant. The removal of load is considered as the application of a negative load and the creep function for a negative load is identical to the creep function of a positive load. Obviously, if secondary creep or reversible changes of phase are part of the creep deformation, the principle is not expected to apply. Leaderman modified the principle by showing that it may be applied to polymers exhibiting nonlinear response to stress if the stress-dependency of the creep function is taken into account. Though it is a simple principle, it is often difficult to visualize all of its implications in the experimental testing of high polymers. Two of these implications were employed by Leaderman to determine whether the primary creep of any polymer may be represented by the superposition principle. The first consisted of calculating a single creep curve from a series of short-time cyclic tests in creep and recovery. If the principle applies, the calculated curve will be identical to an actual experimental curve. Secondly, if the superposition principle applies, the creep and recovery curves of long-duration tests will be identical over most of the experimental time interval. The latter is a convenient test of the ideality of primary creep response.

THE RELATION BETWEEN MECHANICAL BEHAVIOR AND STRUCTURE

The problem of relating mechanical behavior and structure in high polymers, essentially, is one of defining and interpreting the mechanical behavior in terms of molecular mechanisms of response to stress. Rigorous interpretations of this kind are exceedingly difficult and frequently recourse is made to the use of analogies in the form of mechanical or electrical models. In either case, some prior knowledge of structure is required if the analog or mechanism of response is to be meaningful.

Molecular mechanisms of response may be characterized by their rate and equilibrium behavior. It is generally simpler to analyze equilibrium behavior than rate phenomena; hence, attention has been directed largely toward equilibrium effects. Unfortunately, the fund of scientific knowledge relating prerupture response to molecular structure in polymers is small, and the complete viscoelastic behavior of a material can be mapped out by one or more mechanical tests without furnishing the information necessary to correlate mechanical behavior with structure. The problem is complicated by the numerous variations in mechanical behavior which occur with often unmeasured variations in specimen preparation conditions, previous mechanical histories, etc. Also, it is not possible with present methods to define molecular structure by other independent methods. Experimentally, the problem is attacked by relating changes in mechanical behavior to known or controlled changes in structure. It is often useful to measure changes in thermodynamic and physical properties as a function of strain and initial polymer structure. The following discussion is

intended to familiarize the reader with the rudimentary and generally empirical relationships between mechanical behavior and structure.

A polymer inherits certain of its fundamental mechanical properties from its monomer unit. Gehman (19) indicates that the forces between polymer molecules will influence mechanical behavior. These forces will be of the same order of magnitude as in the monomer except for modification due to enhanced rigidity and orientation of the molecular chains. He points out that the intermolecular forces will be weakened by an increase in temperature, but that the temperature dependence has not been well defined for most polymers. As a general rule, an increase in temperature merely shifts the response to stress toward earlier times if the mechanisms of response are due to changes in configurational molecular structure. Increased molecular activity at higher temperatures increases the probability that a molecular segment will surmount its energy barrier and jump to a new position. Polymers are often characterized by calculating the apparent activation energy related to configurational elasticity from the temperature dependence of response to stress (3). Mark (20) calculated the molar cohesions per unit of chain length for a number of polymers. The molar cohesion of cellulose is high. It is comparable in magnitude to the molar cohesion of the synthetic polyamides, and approximately five times greater than the molar cohesion of rubber.

The symmetry of packing of the cellulose chains influences the response to stress. It is dependent on the molecular cohesion, the ease of kinking and curling of the molecular chains, and variables in the preparation of the polymer. Highly kinked and curled molecular configurations occur when rotation about single valence bonds in the polymer chain is free and

uninhibited as in rubber. Materials of this type are highly extensible. The cellulose molecule lacks flexibility due to steric hindrance which prevents free rotation about the oxygen bridges (21). In cellulose, therefore, the amount of response attributable to changes in molecular configuration is small.

Alignment of the molecular chains may result in crystallization under normal conditions if the intermolecular forces are high enough to counterbalance the thermodynamic driving forces tending to maximize the configurational randomness. As a first approximation, the problem of crystallization in polymers is the same as crystallization in low molecular weight materials. Complete crystallization in polymers is never obtained because of interference between crystallites competing for the same amorphous material. Crystallization is an equilibrium phenomenon. Changes in external conditions, the application of an external stress, etc., may shift the equilibrium to a new position. Changes in phase are common in rubber. Crystallization begins at relatively high extensions when the molecules are uncoiled sufficiently to allow the intermolecular forces to complete the process of alignment. Prior to crystallization in rubber, part of the deformation is due to viscous flow. After crystallization begins, viscous flow no longer occurs and further deformation is reversible. Field (22) investigated the creep properties of rubber in the range where crystallization occurs. He found that before crystallization an increase in stress increased the rate of response, but that as crystallization started, the rate of creep approached a maximum value. In the stress-strain curve, crystallization of rubber corresponds to the flat or readily extensible portion of the curve.

As increasing portions of the polymer become crystalline, the elastic modulus increases and the stress-strain curve shows an upward inflection. A significant upward inflection does not occur until considerable portions of the polymer are crystalline and could occur in the absence of crystallization; hence, the upward inflection is not an adequate test of crystallization. In rubber, the percentage of crystalline material may increase from zero to almost complete crystallinity. Holt and McPherson (23) have established that the crystallization of rubber by external stress is subject to hysteresis and that the melting process is more rapid than crystallization when the changes occur at various constant lengths. In most other polymers, the phenomenon of crystallization has not been investigated as thoroughly. Polymers with high retardation times, for example, may exhibit complex crystallization effects. The stress-induced crystallite growth may be permanently or temporarily nonrecoverable if the intermolecular cohesion is high. If changes in phase during straining of high polymers are suspected, it becomes advisable to establish those changes by measurement of changes in specific volume or by x-ray studies.

Deformation-stress-time relationships are useful in relating mechanical behavior to structure if the presence or absence of phase changes can be established by independent methods. The most thorough attempts at interpreting creep behavior in terms of molecular mechanisms of response have been made by Press (13) for viscose rayon, Press and Mark (14) for acetate rayon, and Leaderman (3) for viscose rayon, acetate rayon, silk, and nylon. Press and Mark point out that the creep curves of many high polymers can be described over fairly long periods of time by a linear relation between

deformation and the logarithm of time. In the case of acetate rayon, the logarithmic creep deformation was partially recoverable and was interpreted as retarded elastic deformation plus crystallization or at least strong interaction between parallelized molecular chains.

Steinberger (24) noted that an equation relating deformation to the logarithm of time would describe the creep curves of cotton fibers.

The slopes of the straight lines on a semilogarithmic plot showed sudden changes from time to time, usually toward lower values. The stress-strain curve of cotton fibers was reported to be linear between stresses of 1 to 27 kg./sq. mm. with a modulus of 530 kg./sq. mm. (25).

During straining of a polymer, work is done on the system and the free energy increases. The increased free energy may be due to either or both an increase in energy or a decrease in entropy. Changes in molecular configuration may be solely an entropy effect. In many polymers, however, changes in internal energy accompany changes in molecular configuration. In rubber, the early extension is an isothermal process and the retractive force of a strained specimen is due almost entirely to the entropy component. As crystallization begins, the change in internal energy becomes significant. The nature of the retractive force in stretched polymers is studied by measuring the changes in tension at constant length with changes in temperature. Wakeham and Gerrow (26) have reported that the entropy contribution to the retractive forces of wet cotton and wet viscose rayon is predominant.

THE VISCOELASTIC PROPERTIES OF PAPER

Investigations of the viscoelastic properties of paper are of rather recent origin and the entire field is in a state of development and consolidation at the present time. Rance (27) has reviewed much of the literature concerning the viscoelastic (rheological) properties of paper through the year 1952. He points out that there are many kinds of paper as a result of a wide variety of fibrous materials and a multitude of processing variables; hence, paper includes materials of widely varying mechanical properties and structure. Unfortunately, Rance does not distinguish with sufficient clarity between experimental fact and the many different and often controversial hypotheses. This difficulty, however, is widespread in this field and apparently stems from speculative interpretations of the mechanical behavior of paper in terms of fundamental and general mechanisms of response to stress. Gibbon (28) states, "Paper must be regarded as a more or less open network of cellulose fibers and other substances and would not be expected to obey recognized laws of elasticity under stresses unless those stresses come into play on the fibers themselves." In essence, Gibbon's statement implies that while one may apply a tensile stress to paper, the stresses on the fibers may be of many types, and the deformation may include a macroscopic reorientation of the fibrous elements in the sheet.

Stress distribution effects in paper may be of vast importance in contrast to polymers of solid cross section. Van den Akker (29) postulates that the response to stress in paper must be essentially the accumulated response of the fibers with due consideration for the distribution of stress throughout the sheet. He suggests the development

of a special statistical mechanical approach to the problem of relating the response of the sheet to the response of the fibers. He argues that it is unreasonable to treat paper as an entity when relating mechanical behavior to structure.

The earlier experimental investigations in this field are attributable to Gibbon (28) and Farebrother (30) in 1944. Gibbon published load-deformation curves of paper and pointed to the yield stress or yield zone of stress as the chief characteristic of those curves. At low stresses, paper responded almost elastically, whereas beyond the yield zone, the specimens were more readily extensible. Rather substantial nonrecoverable deformations were noted in the first loading and deloading; however, following this first test, the response was more nearly reversible. A load-unload hysteresis was noted. A mechanism of fiber slippage was advanced to account for the nonrecoverable deformation. Farebrother extended the work of Gibbon to board and observed similar effects. His principal conclusion was that a correlation between prerupture response and ultimate strength seemed probable.

In 1947, Steenberg (5) presented the first of a series of papers by Swedish workers regarding the load-deformation properties of paper. It was emphasized that the nature of the load-deformation curve was dependent on the previous mechanical treatment of the specimen, orientation of the specimen in respect to the paper machine, relative humidity, and rate of straining. A primary creep curve was obtained on a mechanically-conditioned specimen and it was concluded that paper, if mechanically conditioned, obeys Boltzmann's superposition principle in short-duration multiple-cycle tests except for the early part of each cycle. In a later paper (31), Steenberg introduced the "microcreping" concept to

account for the nonrecoverable deformation of paper. It was proposed that a superficial invisible creping or kinking of the fibers is present in an unstrained sheet, which is removed during straining. In a later paper (32), the concept was amended to the degree that all of the deformation of paper was regarded as ultimately reversible in view of the fact that it cannot be proven to be permanent.

Ivarsson and Steenberg (33) attempted to fit the response of mechanically conditioned paper to the theory of Halsey, White, and Eyring (11). Reasonable qualitative agreement with the theory was obtained; however, unless the dimensions of the flow unit and the apparent energy of activation for flow, which are determined for paper, are checked for papers of different sheet structure at varying external conditions and found comparable to predicted values, the fit must be regarded as a first approximation.

Rance (27, 34) disputes the hypotheses of Steenberg, et al., and proposes that the nonrecoverable deformation of paper is a consequence of the rupture of fiber-fiber bonds at an ever increasing rate until final termination of the test by rupture. The time dependency of the deformation was attributed to frictional sliding of fiber over fiber plus possible additional elastic contributions due to redistributed stresses during straining. Dimensional recovery by wetting is visualized by Rance in terms of the possible reformation of the broken bonds. Rance points to a series of creep curves as evidence of the bond-breaking theory. These curves were flat at early times on the semi-logarithmic plot, but rose sharply in deformation at longer times of

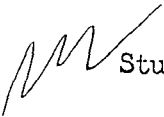
loading. The sharp rise in response was accepted as an indication of a progressive rate of fiber-fiber bond rupture. Prior to the upward curvature, the shape of the curves suggests a period of decreasing rate, however. Further evidence in support of this concept was drawn from the fact that the breaking load of paper in tension decreased linearly with the logarithm of the time of its application. This behavior was reported by Rance (34) and Jacobsen (35). It should be pointed out that the bond ruptures visualized by Rance are between formerly discrete elements of fibrous structure, and are not to be confused with the breaking and reformation of secondary bonds within the solid fibrous structure which normally can occur with changes in molecular configuration. The chief point of contention in Rance's hypothesis is whether fiber-fiber bond rupture can be considered as a rate-controlling mechanism of deformation.

An increase in the specific scattering coefficient with deformation was demonstrated by Nordman, Gustafsson, and Olofsson (36) and was accepted as evidence of internal damage to the sheet during straining. Haward (37, 38) has shown that internal damage occurs in cellulose acetate subjected to tensile loads. This was indicated by a decrease in tensile strength with time of loading and by an increase in opacity. Haward demonstrated, however, that most of the nonrecoverable deformation was actually recoverable if the temperature of the specimen was increased. Despite the recovery of length, the tensile strength was permanently decreased.

Maynard (39) reported that paper subjected to strain in the post-yield range of load-deformation tests will increase in thickness by as much as 10%. He attributed this behavior to the rupture of fiber-fiber bonds.

Mason (40) felt that macroscopic movement of fibers in the sheet was a reasonable mechanism of deformation. Mason did not specify whether fiber-fiber bond ruptures were necessary for this type of deformation.

An excellent historical review and discussion of the mechanical model analogy is given by Leaderman (3). The application of the mechanical model analogy to paper was introduced by Steenberg and others (5, 31, 33) following similar applications to textile fibers by Halsey, White, and Eyring (11). The use of mechanical models to represent the first response of paper to stress was explored by Andersson, Ivarsson, Nissan, and Steenberg (32). An "apparent viscosity" of the fluid in the dashpot was calculated from load-deformation data. This "apparent viscosity" was found to be stress dependent at lower stresses, but approached a limiting minimum value which was offered as a useful single-valued parameter in describing the viscous properties of paper. Recently, Andersson (41) discussed further the characterization of paper by the parameters of mechanical models.

 Studies of the effect of strain rate on the load-deformation curve by Andersson and Sjöberg (42) indicated that initial slope was not affected by rate of strain and that the deformation at rupture was not affected significantly. The slope of the final straight-line portion of the curve and the stress at rupture were increased slightly with increased rates of straining.

The effect of temperature and moisture content on load-deformation properties was studied by Andersson and Berkyto (8). Rather sudden breaks in the response versus moisture content curve occurred between

relative humidities of 60 and 70%. The breaks were greatest for machine-direction specimens and corresponded to a jump in response with increasing relative humidity. The initial and final slopes of the load-deformation curves decreased markedly with increasing relative humidity. It proved difficult to describe the behavior with simple empirical equations. The effect of temperature was investigated only at 0% R.H. Increasing temperature caused a reduction in tensile strength and apparent elastic modulus. The response to stress increased as shown by an increase in deformation at equivalent values of load in constant strain-rate tests.

Ivarsson (9) studied the mechanical conditioning of paper in cyclic load-deformation tests. Numerous cycles were required before the response approached reversibility although the bulk of the nonrecoverable deformation occurred in the first cycle. It was noted that after several cycles of loading and unloading between any two given loads, a continuation of the load-deformation test raised the level of the curve compared to a continuous test. This was attributed to an orientation of the fibers in the direction of the strain, presumably as a result of the strain. Actually, if a straightening and subsequent orientation of fibers is responsible for the increased level of load in the resumed load-deformation curve, the fiber straightening is not proportional to deformation. These data indicate rather that greater intermittent time under load may be the causative factor. An upward rise in the final slope of the load-deformation was noted in many tests and was attributed to the same mechanism.

Arlov and Ivarsson (43) reported that increased tension during the drying of paper will result in increases in apparent elastic moduli and considerable increases in the level of the load-deformation curve. This

effect was interpreted in terms of a reduction in "microcreping." It was proposed that the major effect of drying tension lies in less kinking and curling of the fibers, and therefore, provides for less response due to straightening of these kinked fibers in mechanical tests. The chief effect of drying tension occurred below 25% moisture content. Wahlberg (44) noted that higher drying tensions caused a reduction in tearing strength and that the drying tensions effect was greatest between moisture contents of 10 to 40%.

Edge (45, 46, 47) studied the effect of drying tension on the tensile strength of paper. He noted that the tensile strength increased markedly with increased tension. Sapp and Gillespie (48) reported similar effects in drying sheets under tensions high enough to increase the specimen length. If the stretching was carried too far, a reduction in strength occurred. It can be assumed that an increase in tensile strength will correlate generally with reduced prerupture response at constant load and time. The explanations for this behavior range from reduced "microcreping" (43), an alignment of fibrils at the fiber surfaces (47), to a better stress distribution between the fibers in subsequent mechanical tests (29). Smith (49) felt that the shrinkage in fiber diameter will not be denied in sheets dried without restraint, and, therefore, because of interfiber bonding, the fibers will be corrugated along their lengths. There is much contradiction in this and other factors relating to the structure and mechanical properties of paper. With few exceptions, the most popular concepts provide for macroscopic changes in sheet structure during straining to account for the nonrecoverable deformation.

Recently, Kubat (50, 51) studied the stress-relaxation properties of paper at varying levels of deformation and stress reached by load-deformation tests at varying rates of straining. It appeared that the stress would relax to varying finite values, but that the relaxation of stress would continue over very long periods of time. The rate of stress relaxation was dependent on the manner of arriving at the stress and deformation corresponding to zero time in the stress-relaxation test. This presented a complexity in the interpretation of the results, since at least part of the stress relaxation behavior in a given test is a function of the structural changes which occur before the start of the test. It becomes more difficult to relate stress relaxation behavior to the initial structural characteristics of the sheet.

This short review of the more pertinent literature regarding the prerule mechanical properties of paper has been presented with some emphasis on the mechanisms of deformation which have been proposed to explain the mechanical behavior of paper. The controversial and often diametrically opposite views demand special care in studying the published experimental data.

APPARATUS

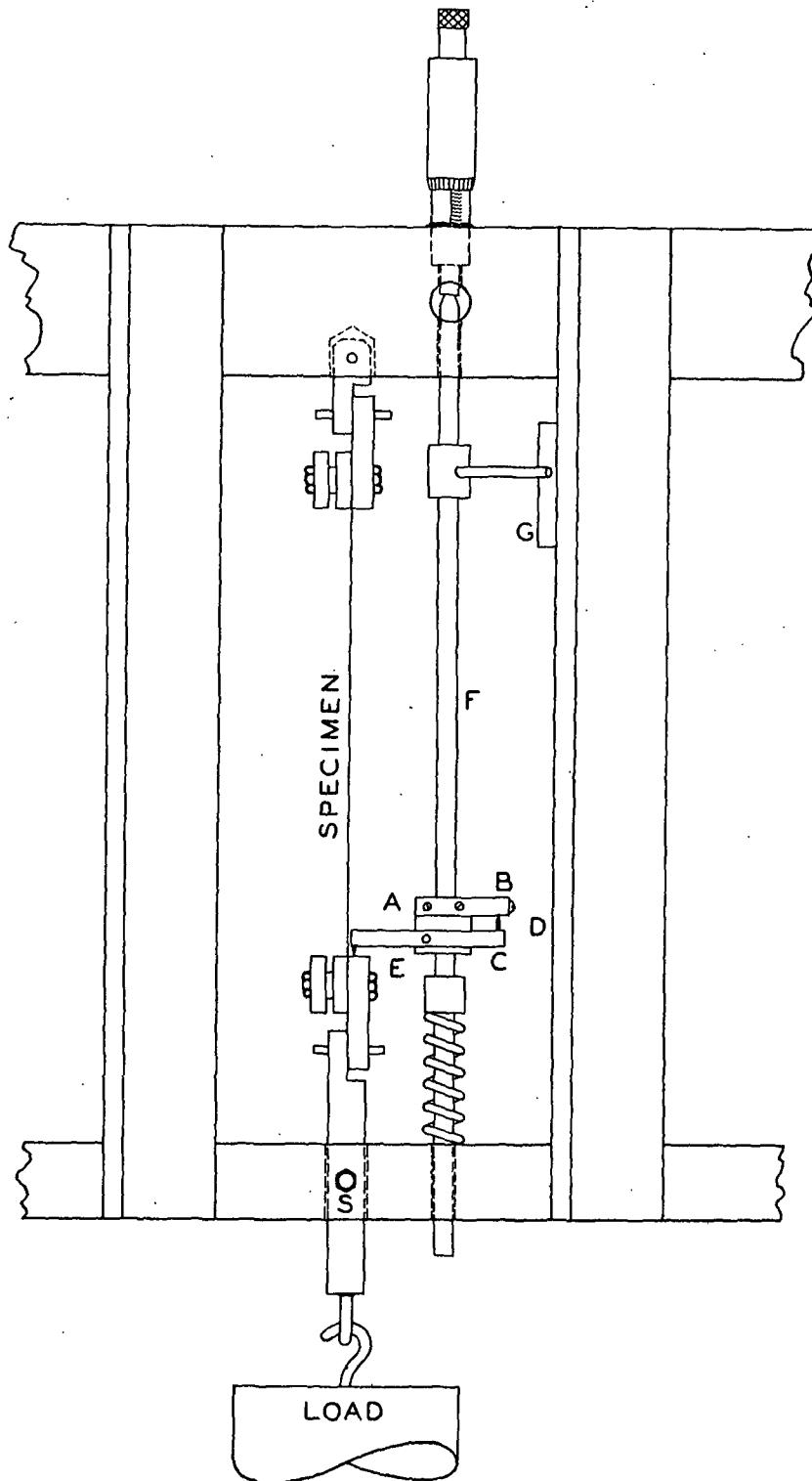
CREEP TESTING APPARATUS

A creep testing apparatus was designed and constructed specifically for use in this study. Dead-weight loading was employed. The upper clamp was fixed and the movement of the lower clamp was measured directly with a micrometer using an electrical contact device to detect the position of the bottom clamp. A schematic drawing of a single creep testing unit is shown in Figure 1. Six of these units were mounted on a single frame. Eighteen units were constructed for this study.

A pair of identical clamps were constructed for each unit. They were removable from the assembly to facilitate insertion of the specimens in the clamps. Each clamp was pinned to a short section of 1/2-inch diameter rod milled flat at one end as shown in Figure 1. The upper rod fitted freely into a hole in the bottom of the top support bar and was pinned in position. The two top clamp support pins are both of 1/8-inch diameter and are perpendicular to form a universal joint which permits any necessary alignment of the top clamp in the direction of the load. This insures uniform loading of the specimen. The lower clamp rod passes through a hole in the bottom support bar. An eyelet attached to its lower end receives a hook attached to the load. The entire lower clamp assembly was balanced about the center line of the specimen and its weight was adjusted to 300 grams. It may hang freely from the specimen or it may be supported by means of the set screw (S) if it is desired to free the specimen of external load.

A micrometer head was soldered into a hole at the top of the support bar approximately 1-3/8 inches from the plane of the specimen. A 1/4-inch

FIGURE 1
CREEP TESTING UNIT



diameter shaft (F) was mounted vertically as an extension of the micrometer screw. It was spring loaded to maintain contact with the screw. Rotation of this extension shaft was prevented by a small horizontal pin which slides against an edge of a guide block (G) attached to the frame. It is held in that position by a twist in the compression spring. Vertical movement of the extension shaft is provided and measured by the micrometer within a range of 1 inch.

An electrical contact device was mounted on the extension shaft opposite the bottom clamp. This device consists of three main parts. The body block (A) fits tightly on the extension shaft and is fastened in position by two set screws. A brass block (B) was fastened to the body block and is insulated electrically from that block. An unbalanced lever arm (C), 2-inches in length, has a pin fulcrum which fits into a horizontal hole in the body block. The lever arm may rotate in a vertical plane. A needle at both ends of the lever arm point in opposite vertical directions. The needle (D) is normally in contact with the polished bottom surface of block (B). In this position, it completes an electrical circuit between the block (B) and the frame. The needle (E) is in position to make contact with the top surface of the lower clamp. Rotation of the micrometer moves this assembly vertically. As contact is made with the lower clamp, an electrical circuit through needle (D) is broken which is indicated by a milliammeter. A current of about 0.5 ma. was employed. The micrometer reading at the instant of completion or interruption of the circuit is a relative measure of the position of the lower clamp.

The lever arm is unbalanced by only 1 to 2 grams which has a negligible effect on the lower clamp position. Any change in spatial orientation of

the lower clamp with the application of load was negligible. Deflection of the upper clamp with loading relative to the micrometer was not detectable. The clamps and specimen could be removed from any unit and reinserted with a maximum change in micrometer reading of about 0.001 inches. Relative movement of the lower clamp could be measured with an accuracy equal to that of the micrometer. Each micrometer was checked and found accurate to 0.0005 inches within the range of the screw.

The top and bottom frame bars were clamped between lengths of $1/4 \times 1-1/2$ -inch angle iron struts spaced at 6-inch intervals along the top and bottom frame bars. At the ends of the support bars (not shown), $5/16 \times 2-1/2$ -inch angle iron struts extended downward as legs for support of the frame. One set of units was mounted on cork and bolted to a concrete block. The other 2 units were set in wax directly on the floor. Each set of 6 units was covered with a polyethylene jacket to minimize air currents around the specimens and damp out the more rapid variations in relative humidity during the tests.

APPARATUS FOR CONTROL OF RELATIVE HUMIDITY

One set of 6 units, excluding the micrometers and loads, was enclosed in a cabinet. Tests at relative humidities other than 50% were run in this humidity cabinet using an external air conditioning system. The relative humidity was maintained near desired values by bubbling the air through appropriate saturated salt solutions. A polyethylene bubble cap was constructed for this purpose. It was four inches in diameter and has two rows of $3/16$ -inch holes drilled around its lower circumference. The holes were placed $1/2$ to $1-1/2$ inches below the liquid surface. The top of the bubble

cap was connected to the outlet of the cabinet. About 1 gallon of saturated salt solution was held in a 5-gallon glass jar which allowed sufficient space above the liquid to remove droplets. The suction of the blower was connected to the top of the jar. Air was circulated through the cabinet horizontally. Air travel in this system was from the cabinet through the bubble cap and the solution then to the blower and back to the cabinet. Normally, equilibrium conditions were reached within 2 to 5 minutes. The blower was an enclosed two-stage unit of the vacuum cleaner^s type. It was operated discontinuously at a fraction of its normal speed. The cycle was approximately 1 minute on and two minutes off. This type of operation provided adequate control of relative humidity with a minimum rise in air temperature over ambient room conditions. Discontinuous operation was satisfactory since the rate of vapor loss or gain was small. The entire system was merely allowed to assume the controlled temperature of the room.

GENERAL EXPERIMENTAL PROCEDURES

PREPARATION OF SPECIMENS

A commercial grade of softwood alpha pulp was used as the fibrous raw material in this work. The pulp was received in the form of dry laps and stored at room conditions.

The pulp was refined in a 1-1/2-pound Niarara-type laboratory beater to desired levels of pulp freeness. The following procedure was employed. About 360 grams of pulp (ovendry basis) were torn up and soaked in water for a minimum period of 12 hours. The soaked pulp was charged directly to the beater and the total volume was adjusted to 23 liters. The initial temperature was adjusted to 73°F. The laps were defibered in the beater without load on the bedplate. After complete defibering, a load of 5500 grams was applied to the bedplate lever arm, and beating continued uninterrupted for a predetermined interval of time. At the completion of the beating, the pulp slurry was diluted to approximately 1% consistency. Schopper-Riegler freeness was determined following Institute Method 414. Pulp freeness versus beating time is given in Table I.

TABLE I
FREENESS OF ALPHA PULP VERSUS BEATING TIME

Beating Time, minute	Schopper-Riegler Pulp Freeness, cc.
0	870
25	775
53	620
70	425

All handsheets were formed on a 17-inch square sheetmold fitted with a new 100-mesh twill-weave wire. A head of water above the wire of 14 to 16 inches was used to form handsheets of approximately 40-pound basis weight (25x40-500). The beaten pulp was added directly to the sheetmold without prior agitation or dilution to minimize the formation of tight fiber bundles which formed readily during stirring with propellertype agitators. One to two minutes of vigorous agitation in the sheet mold were required to provide uniform distribution of the fiber throughout the sheet mold. After the wet mat was formed, the drainage valve was closed and a slight vacuum was applied beneath the wire to compact the sheet and permit the application of a couch blotter without disturbing the newly formed sheet. It was necessary to use prewetted couch blotters, since dry blotters buckled severely when placed in contact with the wet sheet. The wet blotter was pressed into contact with the handsheet using a light felt-covered roll. The pressures across the wire were allowed to equalize and dry blotters were applied to reduce the moisture content of the entire assembly. Blotting was continued until the wire was free of excess water and an adequate handsheet to blotter adherence was obtained. At this time, water was introduced rapidly into the leg of the sheetmold to build up air pressure under the wire and free the handsheet from the wire. The handsheet and couch blotter could then be lifted from the wire without damage to the sheet. The two were placed sheet-side down on chrome-plated metal plates.

The handsheets were wet pressed in stacks of 2 to 6 sheets using two dry blotters over each couch blotter. The desired pressure was reached in a period of 30 seconds and maintained for 5 minutes. A single

wet pressing was employed. After pressing, the sheets adhered to the metal plates and the couch blotter could be stripped from the sheet. Each sheet was taped to the metal plate along the edges to promote adherence during drying. Taping was accomplished by applying 1/2-inch wide strips of gummed kraft tape to the sheet along its edges, and 1-inch wide pieces of "Scotch" cellophane tape to the kraft tape and the plate. All handsheets were dried in accordance with TAPPI standard conditions of temperature and humidity. No significant shrinkages could be detected while the strips were on the plate; however, after removal and conditioning for several days, shrinkages of 0.25 to 0.50% were observed.

Each handsheet was trimmed to a 14-inch square. Specimens were razor-cut from the handsheets to 1 x 12-inch dimensions using a steel template. Specimens were cut parallel to each other and numbered with the sheet number and position of the specimen in the sheet. For example, Specimen 23-8 was cut near the center of Handsheet 23 adjacent to Specimens 23-7 and 23-9. This numbering procedure was adopted to permit an assessment of possible variation in creep behavior to possible variability across the handsheet.

CHARACTERIZATION OF HANDSHEETS

Each specimen was weighed at 50% R.H. immediately after cutting to determine individual basis weight. Reported basis weights of the handsheets are averages for the specimens of each handsheet. The average moisture content at the time of weighing was approximately 7.8% (ovendry basis). The moisture content declined slightly with continued storage at 50% R.H. to about 7.4%.

Handsheet caliper was determined by measurement of the thickness of the stack of specimens from each handsheet at three or more positions along the specimen length. Each stack consisted of 5 to 11 specimens. The reported values are the average calipers of individual specimens. The Cady micrometer was used in accordance with the apparatus requirements of Institute Method 508.

Handsheet solid fractions were calculated from the basis weight and caliper assuming an average fiber density of 1.5 g./cc. at 50% R.H. The same density was assumed in calculating the average solid cross-sectional area of the specimens for calculation of the average initial stress in creep tests. This fiber density is acceptable as a means of comparing handsheets and initial stresses and is not presumed to be a precise value.

Measurements of the reflectance R_0 and R_{∞} of samples of the handsheets backed by black velour paper and backed by optically-infinite pads of the same sheet were determined by the General Electric Recording Spectrophotometer at a wavelength of 600 m μ . Scattering coefficients were obtained from these measurements using the Bureau of Standards opacity charts (52). Specific scattering coefficients were calculated by dividing the scattering coefficients by the basis weight of the specimens at 50% R.H. These values are reported chiefly to compare the relative unbonded areas of handsheets prepared from pulp of the same freeness at different degrees of wet pressing.

Four wet handsheets prepared from 620 cc. S.-R. freeness pulp were stacked in the British sheet mold between sheets of filter paper, and two liters of acetone were slowly filtered through the pad to replace the water. The acetone was replaced in a similar treatment with two liters of butanol.

These handsheets, after air drying from butanol, had a specific scattering coefficient of approximately 960 sq. cm./g. Such "butanol handsheets" were not prepared at the other pulp freenesses.

A load-deformation curve was run on a single specimen of each handsheet as an estimate of variability between handsheets to aid in the selection of specimens for creep tests. The tensile strength in load-deformation tests at a constant strain rate of 1% per minute was recorded for 10-inch initial specimen lengths and reported as stress in kg./sq. mm. based on the initial calculated cross-sectional area of the specimens.

Pertinent data relating to the handsheets which were prepared are given in Table II. Only a part of these handsheets were used in the creep tests studies; however, others are listed to provide a better estimate of handsheet characteristics. The single-spaced groups represent handsheets which were pressed at the same time.

Moisture content versus relative humidity data were obtained by the method of Wink (53). Random samples from each group of as-dried handsheets were selected at 50% R.H. and exposed to several progressively lower relative humidities to obtain the desorption data. Each sample was then dried over phosphoric anhydride to obtain the dry weight. Following this drying, the samples were exposed to progressively greater relative humidities to obtain the moisture content versus relative humidity data on the adsorption curve. The relative humidities were maintained by the use of appropriate saturated salt solutions. The samples were exposed to each relative humidity until the change in weight between successive 24-hour weighings was less than 0.2% of the dry sample weight.

TABLE II

CHARACTERISTICS OF HANDSHEETS

Handsheet Number	S.-R. Pulp Freeness, cc.	Wet Pressure, p.s.i.	Average Caliper, mil	Basis Weight, mg./sq. in.	Calculated Solid Fraction, %	Tensile Strength, kg./sq. mm.	Specific Scattering Coefficient, sq. cm./g.
21	585	50	3.10	36.5	47.9	--	--
22	620	50	2.56	31.6	50.1	--	--
23			3.11	36.9	47.3	7.38	--
24			3.09	36.9	48.6	7.75	--
25			3.05	36.7	49.0	7.18	348
26			3.06	36.2	48.1	7.70	--
27			3.10	36.6	48.0	7.61	--
28	620	50	3.29	36.9	45.6	7.41	364
29			3.21	35.6	45.1	6.70	357
30			3.30	36.4	44.9	6.59	--
31			3.31	36.2	44.5	7.00	367
32			3.23	36.4	45.8	7.20	364
33			3.20	36.7	46.7	7.03	362
34	620	200	2.81	36.0	52.1	7.94	--
35			2.79	35.8	52.2	8.03	--
36			2.84	36.1	51.7	7.35	309
37			2.78	35.1	51.4	7.44	--
38			2.79	36.1	52.6	7.94	--
39			2.84	36.4	52.1	7.52	--

Continued on next page

TABLE II (Continued)

CHARACTERISTICS OF HANDSHEETS

Handsheet Number	S.-R. Pulp Freeness, cc.	Wet Pressure, p.s.i.	Average Caliper, mil	Basis Weight, mg./sq. in.	Calculated Solid Fraction, %	Tensile Strength, kg./sq. mm.	Specific Scattering Coefficient, sq. cm./g.
40	620	800	2.62	36.5	56.6	--	262
41			2.63	35.6	55.0	8.44	252
42			2.63	35.1	54.2	8.66	--
43			2.60	37.0	57.8	7.78	244
44			2.93	37.0	51.4	7.58	271
45			2.63	35.8	55.3	8.14	--
46	775	10	5.04	39.2	31.6	4.57	441
47			5.03	39.1	31.6	4.49	436
48	775	50	3.87	38.7	40.6	5.21	412
49			3.90	38.7	40.4	5.35	411
50	775	400	3.03	37.7	50.6	6.07	339
51			2.98	39.1	53.4	6.54	--
52	775 ^a	50	4.08	40.0	39.9	5.44	315
53			4.00	38.9	39.5	5.44	331
54	425	50	3.00	37.6	51.0	8.52	336
55			3.01	38.3	51.7	7.72	336
56			3.03	38.1	51.1	7.88	347
57			3.03	38.3	51.5	7.50	333

^a Fraction of 775 cc. S.-R. freeness pulp retained on 20-mesh screen in Bauer-McNett classifier.

All specimens were aged for a minimum of 14 days prior to testing. Specimens from Handsheets 28 through 33 were tested at intervals throughout the course of these creep studies (50% R.H. and 73°F.) as controls to determine possible changes in creep behavior because of aging. No significant aging effect was noted, however.

CREEP TESTING PROCEDURES

In all tests at the various relative humidities, specimens in the as-dried condition were inserted between clamps at an initial length of 10.00 inches. The initial specimen width was 1.00 inch. The clamps were removed from the apparatus and inserted in a metal jig. A steel spacer separated the clamps and provided for alignment of the clamps and of the specimens in the clamps. The specimens were normally handled at the ends in areas not subjected to testing. Where this was not practical, rubber thimbles were worn over the fingers of the operator to avoid possible changes in the specimen moisture content history. The clamps and specimens were inserted into the creep testing apparatus with the lower clamp assembly supported so that each specimen was completely free of external load until the start of the creep test.

At the start of the creep test, the initial micrometer reading was made with the 300-gram load on the specimen (weight of the lower clamp assembly). The measured total deformations in both creep and creep recovery tests were corrected by addition of the small deformations due to the 300-gram load. These small deformations were estimated by measuring the small reversible deformations upon application and removal of 100 to 500 grams of additional load. These measurements are not accurate,

principally because creep effects may be substantial even at these small loads. These data were used, however, for estimating the apparent elastic modulus with an accuracy of $\pm 10\%$ or better depending on the magnitude of the apparent elastic modulus.

The creep load was applied manually to the freely suspended lower clamp assembly within a period of less than 0.5 second. Time was recorded in seconds from the instant of load application, or removal in the case of creep recovery tests. The first readings in creep tests could be obtained at about 10 seconds of elapsed time. The customary procedure for obtaining a reading involves driving the micrometer beyond the end point to break the electrical circuit. The instant at which the circuit was completed is indicated by the milliammeter. Time is recorded at that instant to the nearest second at early times and to four significant figures at longer times. At lower rates of creep, readings were taken by driving the micrometer extension shaft downward until the circuit was just broken.

In obtaining creep recovery curves, the load was removed rapidly and the lower clamp assembly was supported so that the specimen was free of external load. If desired, creep recovery curves could be obtained with varying amounts of residual load on the specimen although no-load recovery curves were customary. Readings in recovery tests were taken by lowering the lower clamp assembly for a period of about 10 seconds. The first readings in recovery tests were obtained at about 100 seconds to minimize the effect of the small clamp load on recovery response.

The deformations in either creep or recovery tests were always measured relative to the specimen length just prior to the application or

removal of load, but were reported with negligible error as a percentage of the initial 10-inch specimen length. Total creep or recovery deformation includes the immediate elastic deformation. The same additive correction was used for creep and recovery tests since any possible changes in these values after deformation had occurred were seldom greater than the accuracy with which the values could be measured. In repeated tests on the same specimen, the total deformation relative to the initial length usually was not reported, but can be calculated easily from the deformations of preceding tests. Creep represents an extension, and recovery, a contraction in length. In reporting recovery deformations, however, the negative sign was not used and both are considered as positive deformations.

FIRST-CREEP PROPERTIES OF ALPHA PULP HANDSHEETS

First-creep curves are those obtained on specimens which have not been subjected to previous mechanical tests. In paper, the first-creep properties will be dependent on the mechanical and moisture-content histories of the specimens. The previous mechanical history of the handsheets used in this study is due largely to the planar restraint of shrinkage during drying. The moisture content history can be considered as primary desorption to the test conditions.

Handsheets which have been dried under planar restraint may exhibit planar shrinkages if subjected to wetting and redrying without restraint. In this sense, the handsheets may be said to possess residual strains, which presumably may vary in magnitude with the shrinkage tendency of the wet fibrous mat and the amount of restraint which was provided during drying. Since it has not been demonstrated conclusively that dried handsheets possess residual stresses, the residual strain terminology will be used.

One seeks in this study to obtain a measure of typical first-creep behavior on specimens which have a minimum of uncontrolled mechanical or moisture-content history. Handsheets were used in this work, since machine-made paper is expected to exhibit more complex creep behavior due to greater heterogeneities in structure and more complex mechanical and moisture-content histories. The objective of this portion of the work is to establish the fundamental relationships between time, deformation, and initial stress in first-creep tests of alpha-pulp handsheets which were air dried to equilibrium at 50% R.H. and 73°F. with essentially zero shrinkage in the plane of the sheet.

FIRST CREEP

A single creep curve describes the relationship between delayed deformation and time within a limited experimental time interval at a single initial stress. A number of creep curves at different initial stresses are required to investigate the effect of initial stress on creep response. The tests should embrace the widest possible range in initial stress and time. The practical ratio of highest to lowest initial stress is limited by rupture at the higher initial stresses and the small delayed deformations at the lower values. This ratio is about 3:1 for these specimens.

Typical behavior in first creep is shown by a series of first creep tests on specimens of Handsheet 23. The tests were run at 50% R.H. and 73°F., at five different creep loads providing a range of initial stress from 2.06 to 6.21 kg./mm.² This range of initial stress is approximately 26 to 84% of the tensile strength in a load-deformation test at a strain rate of 1% per minute. In these, as in all other tests, the initial specimen dimensions were 1 by 10 inches in width and length. Pertinent data relating to Handsheet 23 are summarized in Table II.

A summary of the first-creep tests is presented in Table III. At the two highest loads, 5.5 and 6.0 kg., the tests terminated in rupture. At the lower loads, the tests were discontinued after 10 days (864,000 seconds). The agreement between duplicate tests was excellent except between the two tests at 5.5 kg. load (see Appendix, Table A). A first-creep curve at each load was plotted in Figure 2 as total first-creep deformation as percentage of the initial length versus the logarithm of time in seconds. This is the customary plot to which the shape of creep curves will be referred.

TABLE III

SUMMARY OF FIRST-CREEP TESTS OF HANDSHEET 23

(All tests at 50% R.H. and 73°F.)

Test Number	Specimen Number	Creep Load, kg.	Average Basis Weight, mg./sq. in.	Initial Stress kg./sq. mm.	$\frac{\Delta L}{L_0}$, % (1000 sec.)	$10^4 \frac{\Delta L}{L_0}$	$\frac{K}{S_0}$, sq. mm./kg. $\times 10^4$
20	23-6	2.00	37.0	2.06	0.28	--	--
17	23-9	3.50	37.0	3.60	0.64	24.6	6.83
18	23-10	3.50	37.1	3.60	0.62	23.9	6.64
15	23-7	4.50	37.0	4.63	1.23	32.9	7.11
16	23-8	4.50	37.1	4.62	1.23	33.4	7.25
19	23-13	4.50	36.6	4.68	1.22	32.2	6.88
27	23-1	5.50	36.8	5.70	1.95	39.4	6.91
24	23-2	5.50	36.8	5.69	1.84	--	--
25	23-11	6.00	37.0	6.19	2.52	41.7	6.74
26	23-4	6.00	36.8	6.21	2.53	44.2	7.12

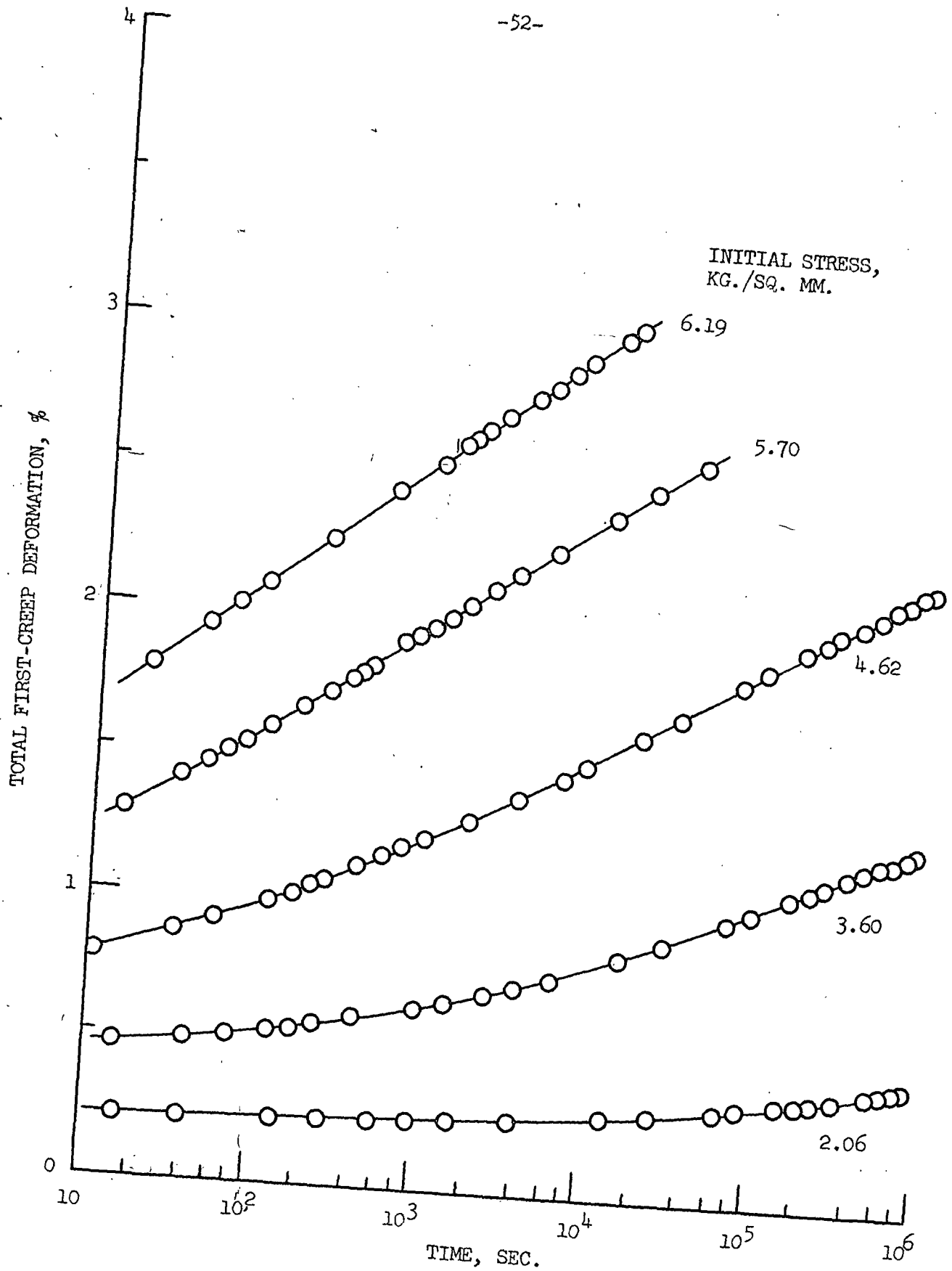


Fig. 2. First-Creep Curves of Handsheet 23

A study of the first-creep curves reveals the presence of at least two kinds of delayed response to stress which are separated in time and joined by a transitional zone of creep. For the purposes of this discussion, a kind of response will be defined by a mathematical equation relating deformation to time at any initial stress within a limited span of time and does not necessarily imply specific mechanisms of response. For example, the creep curve in the first two decades of log time at 3.60 kg./mm.² and practically the entire curve at 2.06 kg./mm.² may be described by an exponential equation,

$$y / L_0 = B t^a + C \quad (1)$$

where y = total first-creep deformation, inches

L_0 = initial specimen length, inches

t = time of loading, in seconds.

and B , a , and C are constants at any initial stress. The exponent, a , was 0.23 at either initial stress. The constant C represents the deformation at zero time and corresponded closely to the immediate elastic deformation calculated from the estimated apparent elastic modulus of 1030 ± 50 kg./mm.² and the initial stress. The constant, C , would therefore be mathematically equivalent to the initial stress, S_0 , divided by the apparent elastic modulus, E_a . The value of E_a calculated from Equation (1) was 1050 kg./mm.² A change in constant B is equivalent to a shift of the exponential curve along the time axis. Creep following Equation (1) will be termed "exponential creep" and occurs over a limited time interval.

Deviation from exponential creep was noted after about 10^3 seconds in the tests at 3.60 kg./mm.² From this point in time to about 10^5 seconds, simple mathematical equations could not be used to describe the relationship

between deformation and time. The response in this region will be termed "transitional." It may embrace two or more decades of log time. Following the transitional zone, creep is linear on the customary semi-logarithmic plot and may be described by a logarithmic equation,

$$\underline{\epsilon} / \underline{L}_0 = \underline{K} \log t + \underline{C}' \quad (2)$$

where \underline{K} and \underline{C}' are constants at a given initial stress. The constant \underline{K} is the slope of the straight line. \underline{C}' has no real significance since it represents the total creep strain at one second where the logarithmic creep equation may not be applicable. Logarithmic creep begins at very early times at high initial stresses and at extremely extended times of loading at the lower initial stresses.

The slopes of the logarithmic-creep portions of the first-creep curves are linearly related to initial stress. The slope versus initial stress curve (see Figure 3) extrapolates to zero slope at zero initial stress. This demonstrates that an essential linearity between this kind of creep response and initial stress may exist over wide ranges of initial stress. The various kinds of response occur at different experimental times at different initial stresses, hence, relationships between total first-creep deformation and initial stress at constant test durations are functions of the test duration. An increase in initial stress appears to have two effects on creep response. First, there is an increase in amount of each kind of response and secondly, a shift of the various types of response along the time axis toward earlier times. A broad experimental time interval is required to observe all three types of response at a single initial stress.

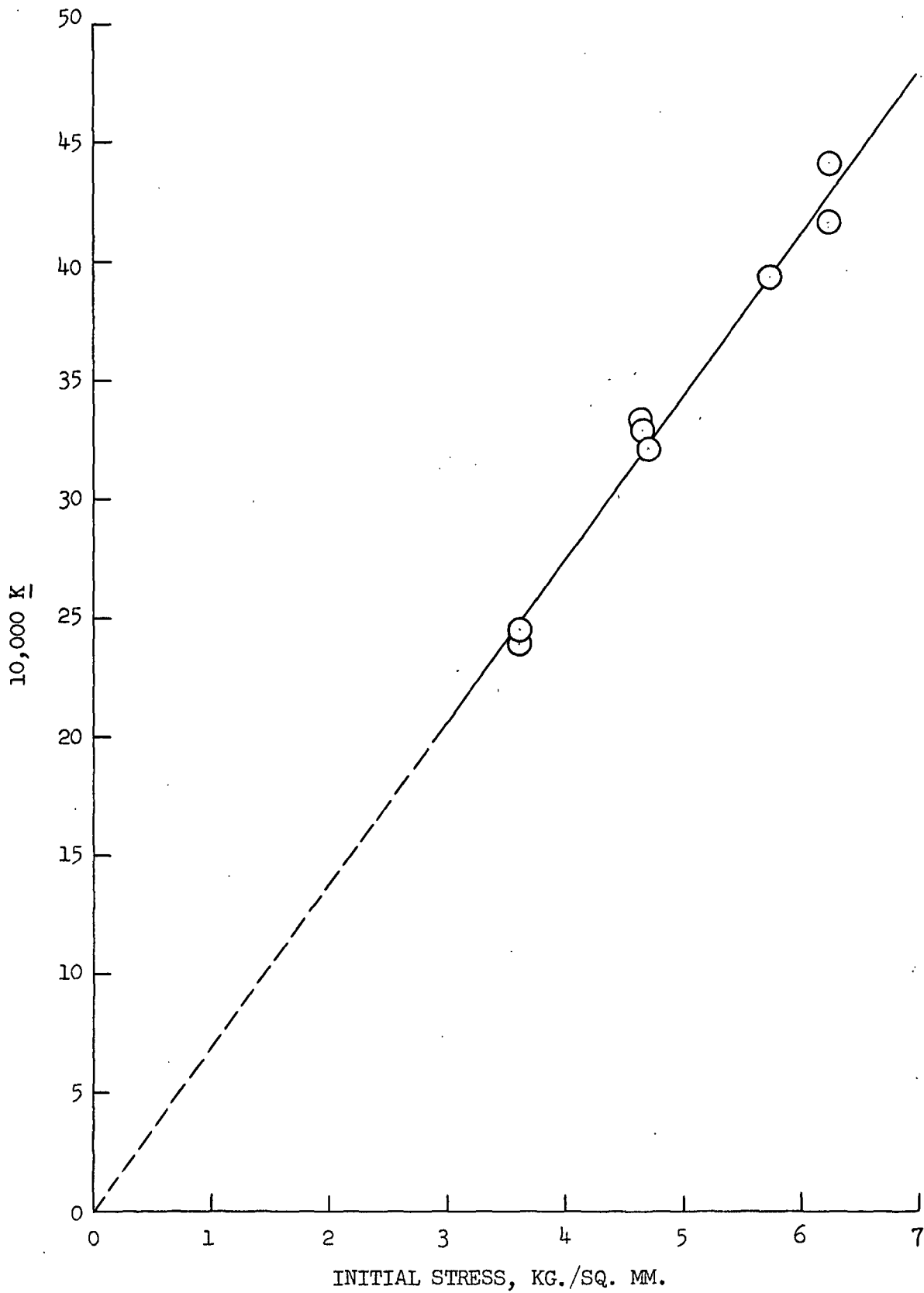


Fig. 3. Constant \underline{K} of Logarithmic First-Creep Equation Versus Initial Stress for Hand-sheet 23

THE MASTER CREEP CURVE

The observed shift of the various kinds of first-creep response along the log time axis with initial stress indicates that a speeding-up of the various mechanisms of response may occur as the initial stress is increased. The concept that one of the effects of increasing initial stress in creep tests of nylon is a speeding-up of the various mechanisms of response has been explored by Catsiff, Alfrey, and O'Shaughnessy (54). It was postulated that a single generalized or master creep curve may describe the total possible response to stress and that the observed response at any initial stress is merely some part of the total. In the case of nylon, they were able to construct a master creep curve by appropriate expansion in deformation of the creep curves at the lower initial stresses followed by shifts of the various curves along both the deformation and time axes until they coincided in the regions of overlap. The authors did not speculate as to the significance of the master creep curve except as it might validate the concept of the speeding-up of response with increasing creep stress.

A master creep curve could be constructed from the first-creep curves of Handsheet 23 by simply reducing the deformations by a factor of initial stress and shifting the reduced curves along the time axis until they coincided in the regions of overlap. The first-creep curves, reduced in deformation by a factor of initial stress are shown in Figure 4. Each reduced curve appears to be a different portion of a total curve. Excellent agreement in the regions of overlap is obtained when these curves are shifted along the time axis (Figure 5). The master creep curve has the time axis of the creep test at 2.06 kg./mm.^2 since that test was

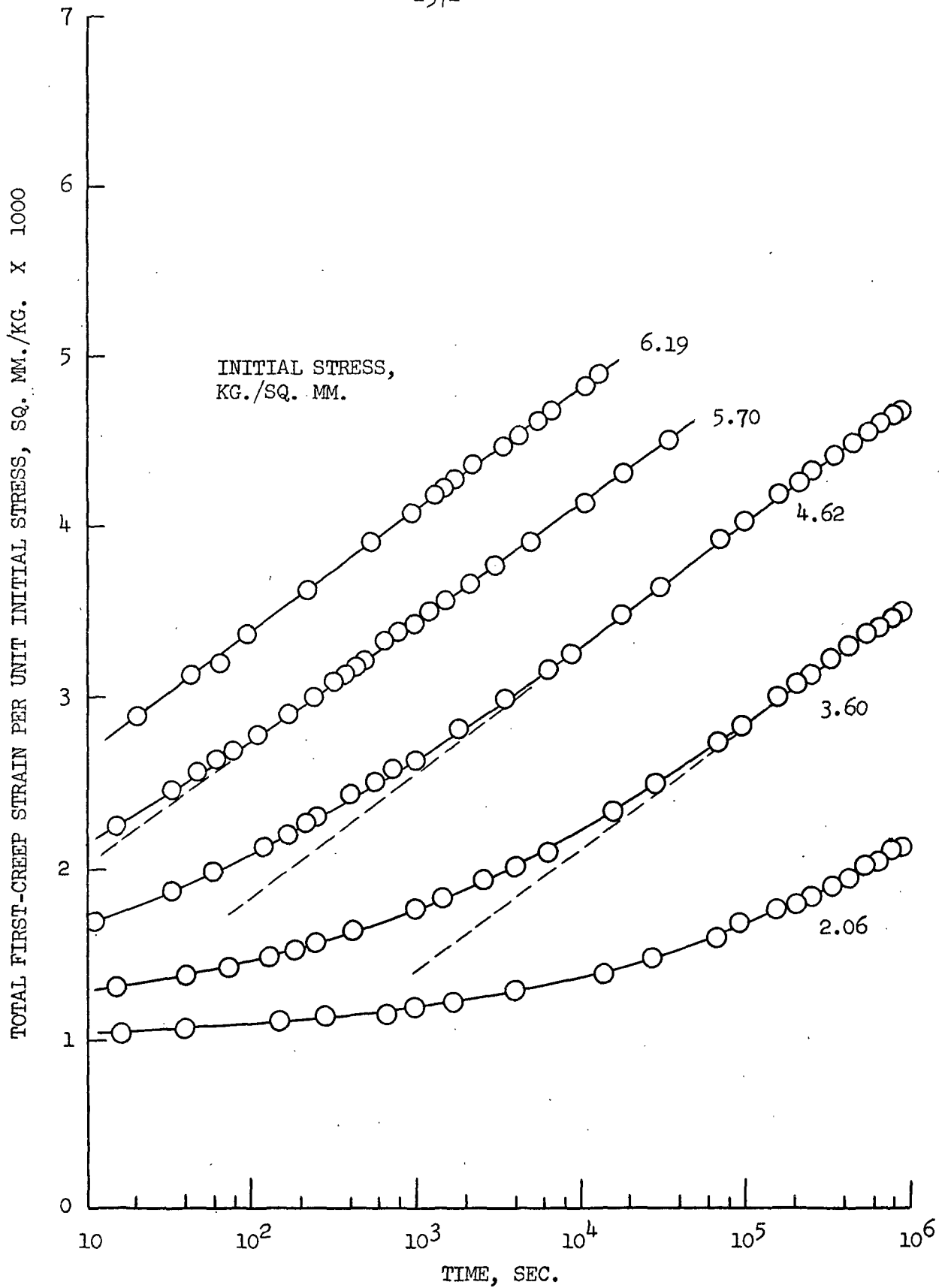


Fig. 4. First-Creep Curves of Handsheet 23 Reduced in Deformation by Factors of Initial Stress

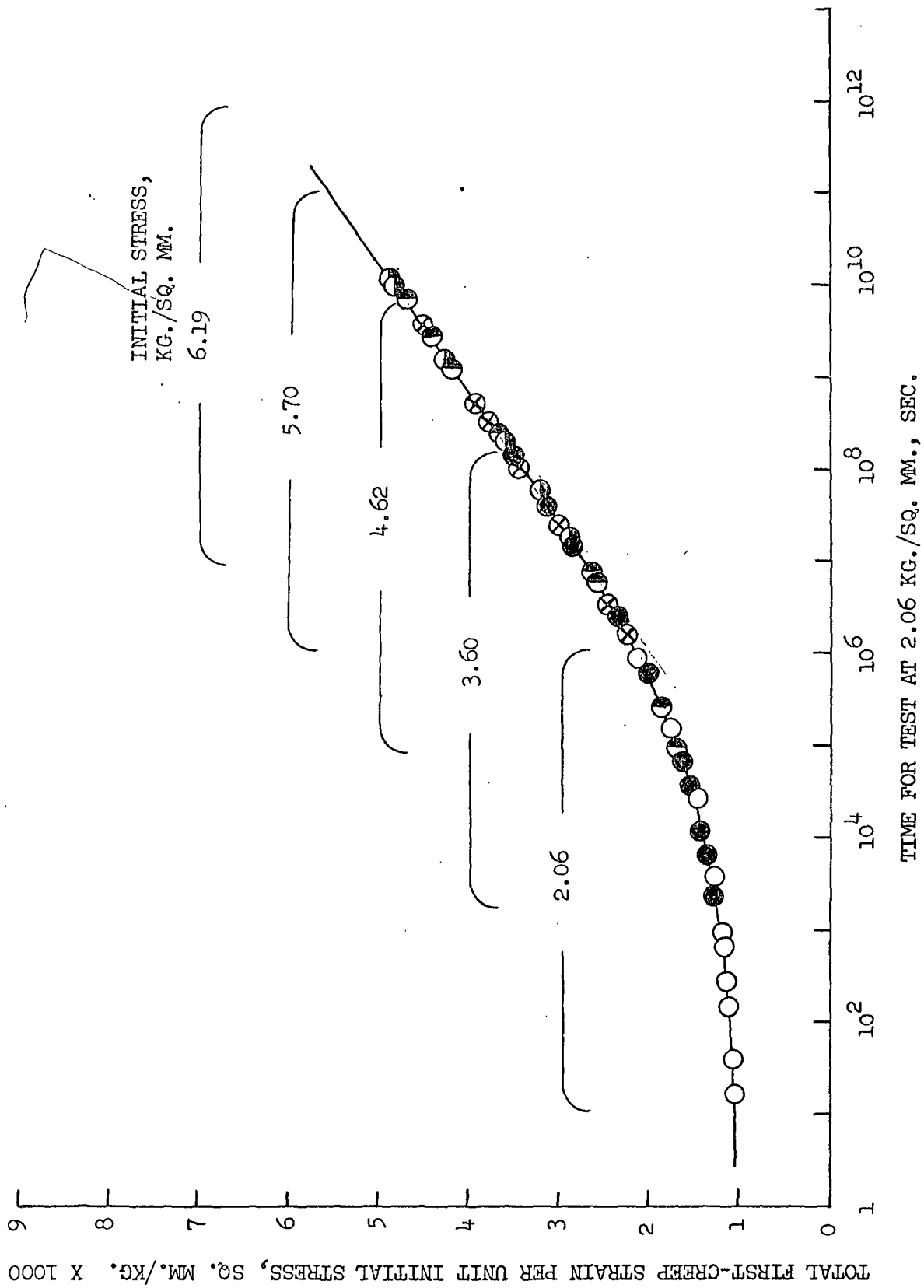


Fig. 5. Master Creep Curve of Handsheet 23

selected arbitrarily as a basis for its construction. The brackets identify experimental time intervals of 10 to 10^6 seconds at each initial stress. The curves at the higher initial stresses were shifted toward greater times to form the master creep curve. These are designated as positive shifts.

The master creep curve can be used as a means of extrapolating the experimental creep curve at any initial stress to earlier or longer times by virtue of the agreement in the regions of overlap. The validity of such extrapolations over very long spans of time cannot be assessed in this work since tests at a single initial stress could not encompass the necessary range in log time. By inference from the master creep curve as a means of extrapolation at each initial stress, one may assume the following:

1. All three types of response, exponential, transitional, and logarithmic, will occur at any initial stress and are linearly related in amount to initial stress although they will occur at vastly different experimental times.
2. The immediate elastic deformation is linearly related to initial stress.
3. The exponent of the exponential equation describing the early portions of the creep curves at any initial stress is independent of initial stress.
4. Logarithmic creep continues for very extended times of loading and occurs at all initial stresses.

5. The slopes of the logarithmic-creep portions of the first-creep curves are linearly related to initial stress at all initial stresses.

THE TIMESHIFT REQUIREMENT IN THE CONSTRUCTION OF THE MASTER CREEP CURVE

The relative shift in log time of the first-creep curves required to form a master creep curve for Handsheet 23 is a linear function of initial stress (see Figure 6). The time-shift requirement, represented by the slope of the straight line, is 1.45 decades of log time per unit initial stress. It is impractical to attempt an experimental determination of the time shift versus initial stress relationship at lower initial stresses because of the small delayed deformations. It was estimated, however, that linearity extends to lower initial stresses as shown by the extrapolation. These estimates are made in the following manner. Curves of total first-creep deformation versus initial stress at various constant times are extrapolated to the origin (see Figure 7). Values of total strain per unit initial stress are calculated at desired initial stresses within the extrapolated portions of these curves. In the master creep curve, the same total strain per unit initial stress is reached at earlier times at higher initial stresses. The estimated strain per unit initial stress at a given time, t_1 , and initial stress S_1 , will fall at an earlier time, t_2 , on a reduced first-creep curve obtained at a higher initial stress, S_2 . In this case, S_2 is 2.06 kg./mm.² The difference between the times, t_1 and t_2 , expressed as a logarithm represents the time shift required to obtain coincidence between reduced first-creep curves at the two initial stresses, S_1 and S_2 . At best, this is a crude procedure, but served to indicate that linearity in the time shift to initial stress relationship may be expected at lower initial stresses.

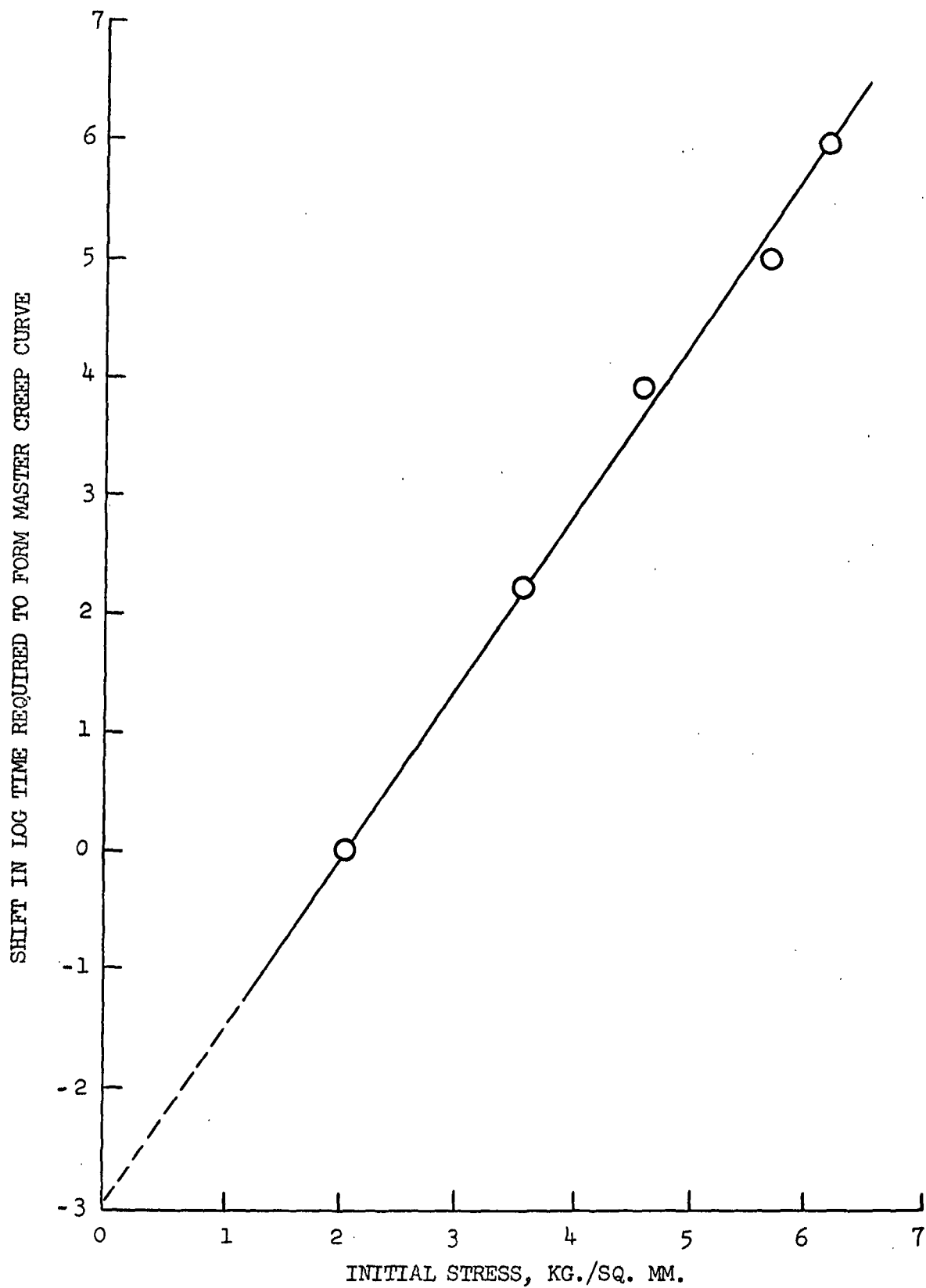


Fig. 6. Time Shift Required to Form Master Creep Curve Versus
Initial Stress for Handsheet 23

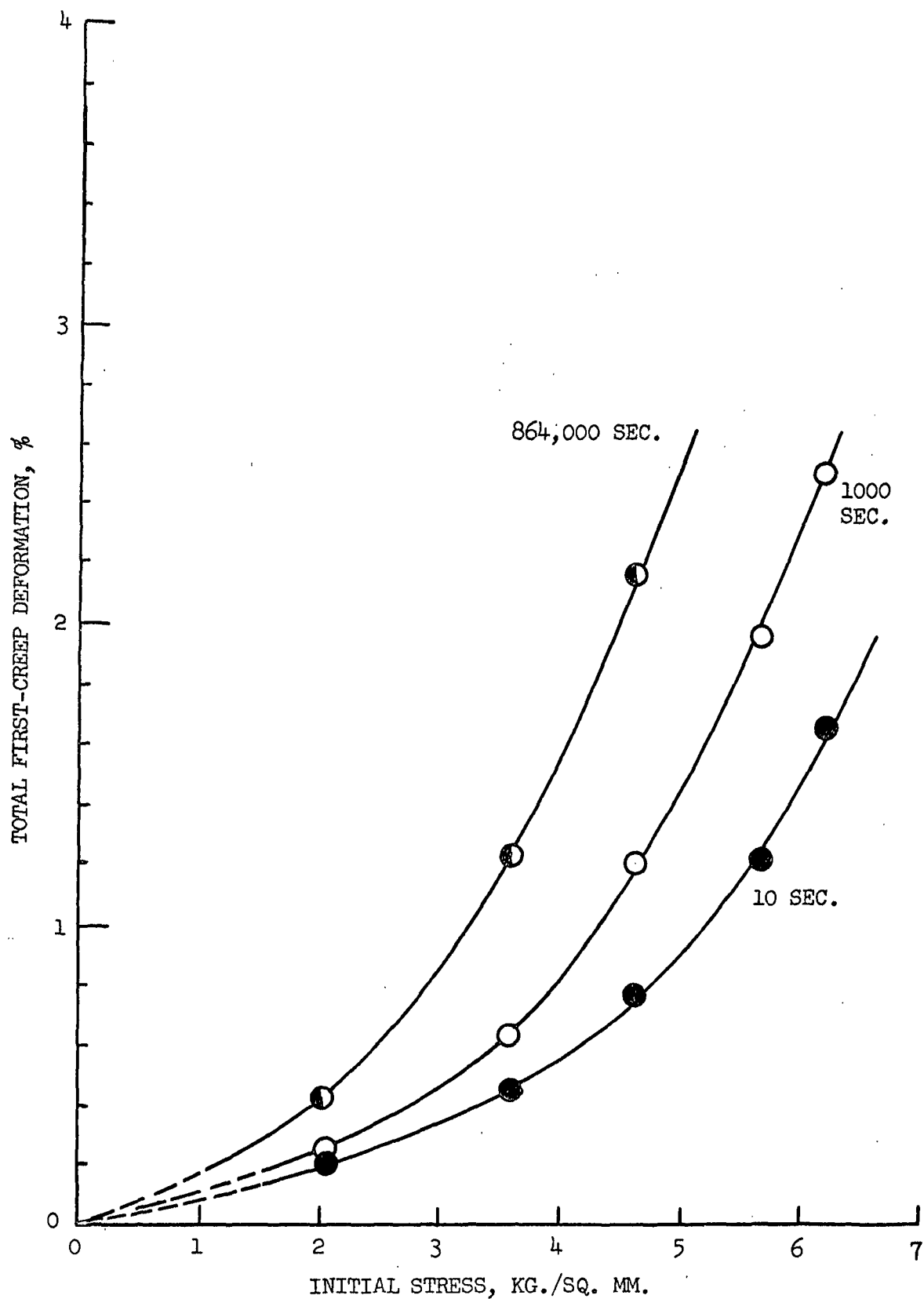


Fig. 7. Total First-Creep Deformation Versus Initial Stress at Various Times for Handsheet 23

An extrapolation of the time shift versus initial stress curve to zero initial stress would imply that a real time axis exists at zero initial stress. This, of course, is a practical impossibility since there can be no response at zero initial stress. Time shifts must be considered only between finite initial stresses.

The time-shift requirement accounts for the nonlinearity between total first-creep deformation and initial stress at constant test durations, but it is not an independent index of nonlinearity. The time-shift requirement is also extremely dependent on the shapes of the first-creep curves. Hence, any significance which might be attached to the magnitude of the time-shift requirement would have to consider the curve shape. It is possible to introduce stress terms in Equations (1) and (2) which are based on the time-shift requirement. This adds little to an understanding of the relationships between the variables of time, deformation and initial stress beyond what has been presented thus far and, therefore, is not described. The effect of increasing initial stress on first-creep response may be described simply as a stress-proportional increase in total deformation plus a stress-proportional shift in log time of the response toward earlier times.

Leaderman (3) concludes that the nonproportionality of deformation to stress must be ascribed to the partially crystalline structure of polymers exhibiting this behavior. It may be indicative of a restraint in the amorphous areas due to the more rigid crystallites. This is hypothetical, since such behavior may be simply a consequence of the high degree of intermolecular bonding in the amorphous areas which invariably is present in partially crystalline polymers.

TOTAL FIRST-CREEP DEFORMATION VERSUS INITIAL STRESS AT CONSTANT TIMES

It is frequently useful to describe creep properties by plots of total creep deformation versus initial stress at constant test durations. Plots of this type for the first-creep of Handsheet 23 are shown in Figure 7. It may be noted that the deformation versus initial stress curves are strongly nonlinear. The shapes of these curves suggest that the viscoelastic behavior of paper might be interpreted in terms of a yield stress or a yield zone of stress, which if exceeded results in easier extensibility. A tendency arises, therefore, to use a yield stress concept in explaining the mechanisms of response to stress. These creep data, however, reveal the stress effect as a continuous process. The two effects of increasing initial stress, namely, the increase in amount of deformation and the shift of the response toward earlier times along the log time axis both act to increase the deformation at constant times of loading. The combined effect is a nonlinear relationship between total first-creep deformation and initial stress. An analysis of the curves of Figure 7 in terms of a yield zone of initial stress is not valid because of the obvious shift in this zone with test duration. The concept of a yield stress is perhaps more tempting when considering load-deformation curves of paper. The same principles, however, will be applicable, but are more complicated because of the predetermined time-controlled variation in either load or elongation common to such tests.

MECHANISMS OF RESPONSE

Mechanisms of response may be either molecular or macroscopic or both. Macroscopic mechanisms of response, however, must be defined by changes

in spatial orientation of macroscopic elements of structure, since ultimately, all response to stress in which deformation occurs in any element of the solid structure must involve molecular mechanisms.

The fact that a simple mathematical equation may describe the relation between deformation and time over several decades of log time is of some importance in predicting the individual mechanisms of response. The mathematical equation is an empirical description of a pattern of response. It has been shown that these empirical descriptions are general at all initial stresses. Consequently, one must assume that the structural changes in creep tests are orderly smooth functions of time. The ability to construct a master creep curve from the first-creep curves at different initial stresses suggests that the pattern of structural change is not affected by changes in initial stress.

These data, however, can give no indication of the number of mechanisms of response which may be associated with a particular function, or of the possible interdependence of the various mechanisms. It is significant, perhaps, that there is a degree of orderliness in the initial stress-time-deformation relationships in first-creep tests, which would not be likely if macroscopic changes in sheet structure accounted for an appreciable percentage of the total first-creep deformation, or if large changes in stress distribution occurred during the course of a creep test. It is possible, though less likely, that changes in stress distribution during the course of a creep test are of the same character at all initial stresses.

The shapes of the creep curves suggest that there may be at least two different mechanisms of response in first creep. In the exponential zone, the creep rate declines least rapidly with time. The transitional

zone represents an area of steadily increasing rate of decline in creep rate. It might be postulated that the early response is due to configurational elastic mechanisms, and that the curve for this type of response is actually sigmoidal with an inflection point somewhere in the transitional zone of creep, but that the sigmoidal shape is not detectable experimentally because of the superposition of another mechanism of response, which is characterized by the logarithmic creep equation.

It appears that the logarithmic function is the limiting type of response to stress in the long-duration regime. No departure from logarithmic creep was noted in Test 29 of Specimen 42-1 from the onset of logarithmic creep at about 3 hours to rupture of the specimen in 48 days. In no case has any deviation from logarithmic creep behavior been noted prior to rupture of any specimen.

It is clear that first-creep curves are of limited value in interpretation in terms of mechanisms of response. Additional techniques of elucidating the mechanisms of response are required, using the empirically-described patterns of response as guides. Methods by which this may be accomplished in part include the determination of the recoverability of successive increments of first-creep deformation, and a study of the effect of moisture content on first-creep behavior. Other techniques, which are often of value, include the measurement of changes in various physical or thermodynamic properties as functions of strain. The latter, however, were not part of this study.

THE RELATIONSHIP BETWEEN CREEP AND LOAD-DEFORMATION TESTS

The load-deformation test is of limited value in describing the mechanical properties of paper, chiefly because of difficulty in interpretation, since either load or deformation is time-controlled according to some predetermined sequence. In creep tests, on the other hand, the variables of time, deformation, and load are separated, providing for easier interpretation of the mechanical behavior of visco-elastic materials. From a practical standpoint, one seeks to define the mechanical properties of a material in their most fundamental form with a minimum of testing. The possibility of relating load-deformation data to the creep properties of paper is of practical interest, since the load-deformation curve is easily obtained compared with the time and effort involved in creep studies. Attention is directed toward the first load-deformation curve which would be relatable to first-creep properties. A study of the first-creep properties of Handsheet 23 indicated that any relationship is likely to be complex.

There are essentially two approaches to the problem of relating creep and load-deformation behavior. These are the graphical and analytical methods. Holloman and Lubahn (55) devised a graphical method for predicting the creep behavior of metals from the results of load-deformation tests at a number of different rates of deformation. Even if their technique were applicable to paper, tests at too many rates of loading would be required to be practical. A graphical approach is time-consuming and, in addition, is unlikely to be accurate since the effect of deformation rate on the load-deformation curve is not large (42).

The derivation of a stress-strain equation from the creep data of plastics has been undertaken by Findley (56) and discussed by Adams (57). Findley postulated that a mechanical equation of state may exist under certain conditions for solids in which the stress at any time in the load-deformation test is a function only of the strain, the strain rate and temperature, and is independent of the path followed in arriving at that given point. He pointed out that a mechanical equation of state concept would be invalid if the specimen structure were altered during the course of the test. This concept in its most rigorous form would not likely be applicable to paper. Nevertheless, it suggests a first approximation approach to the problem of relating creep load-deformation tests.

In first-creep and first load-deformation tests, neither the load nor the deformation decreases with time during the course of the test. A structural condition of state might be defined by the existing load and deformation at constant temperature and relative humidity. Even if irreversible structural changes occurred in the first-creep tests, it might be postulated that load and deformation are sufficient conditions in defining the rate of creep at any instant. This postulate was tested only for Handsheet 23, by comparing the creep rate, deformation, and load in creep and load-deformation tests.

A load-deformation curve (Figure 8) was obtained on specimen 23-5 at 50% R.H. and 73°F., with the Baldwin-Southwark Universal Tester. A constant rate of deformation of 1% per minute was employed. Initial specimen dimensions were 1.00 by 10.00 inches in width and length.

The controlled constant rate of deformation in this test enables the calculation of the time at any point on the load-deformation curve.

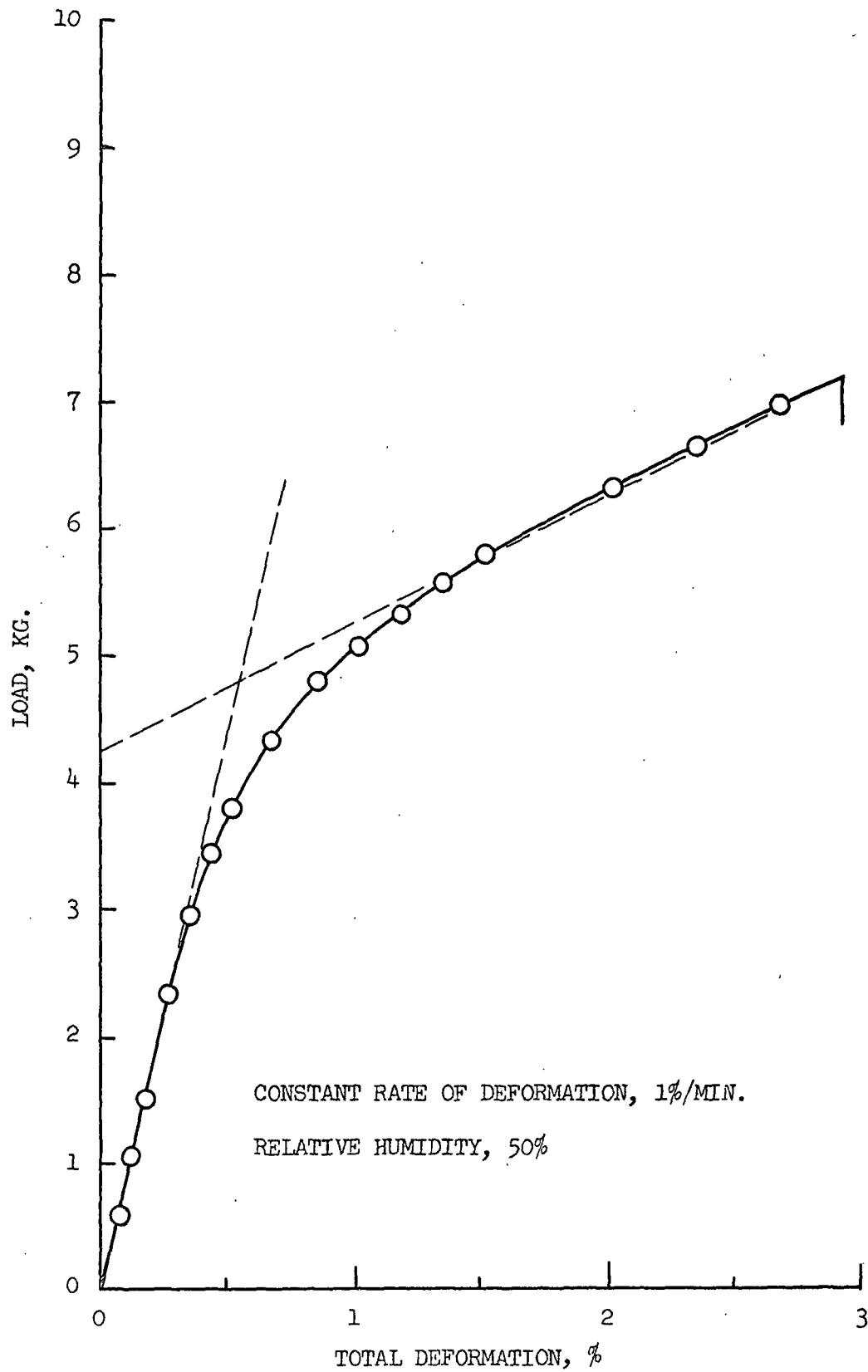


Fig. 8. Load-Deformation Curve of Handsheet 23

It is then possible to calculate the creep rate at any point on the load-deformation curve if the apparent elastic modulus \underline{E}_a is assumed to be constant throughout the course of the test. Any deviation in \underline{E}_a is expected to be small. In this analysis, the differences in initial solid cross-sectional areas \underline{A}_0 of the specimens are negligible, hence, for the sake of convenience these data are analyzed in terms of load \underline{P} rather than initial stresses \underline{S}_0 . The slope of the initial straight line in the load-deformation test, equal to $\underline{E}_a \underline{A}_0$, was 1000 kg. The constant rate of deformation $\underline{dy}/\underline{dt}$ is the sum of two rates of deformation, the rate of creep deformation $\underline{dy}_c/\underline{dt}$ and the rate of immediate elastic deformation $\underline{dy}_o/\underline{dt}$ as indicated in Equation (3).

$$\underline{dy}/\underline{dt} = \underline{dy}_o/\underline{dt} + \underline{dy}_c/\underline{dt} \quad (3)$$

The rate of immediate elastic deformation is equal to the time rate of load increase $\underline{dP}/\underline{dt}$ divided by the product of the apparent elastic modulus and the initial solid cross-sectional area as in Equation (4).

$$\underline{dy}_o/\underline{dt} = (\underline{dP}/\underline{dt}) (1/\underline{E}_a \underline{A}_0) (\underline{L}_0) \quad (4)$$

Substitution of this quantity in Equation (3) gives Equation (5),

$$\underline{dy}_c/\underline{dt} = \underline{dy}/\underline{dt} - (\underline{dP}/\underline{dt}) 1/\underline{E}_a \underline{A}_0 (\underline{L}_0) \quad (5)$$

which is employed for calculation of the creep rates in the load-deformation test. Values of $\underline{dP}/\underline{dt}$ may be calculated by multiplying the slope of the load-deformation curve $\underline{dP}/\underline{dy}$ by the constant rate of deformation $\underline{dy}/\underline{dt}$. In this load-deformation test, however, it was easier to calculate these values from a plot of load versus the logarithm of time (see Figure 9). It is noted that the greater portion of the load-deformation curve may be described with small error by a straight line on this semi-logarithmic plot. The curvature was noted in varying degrees in

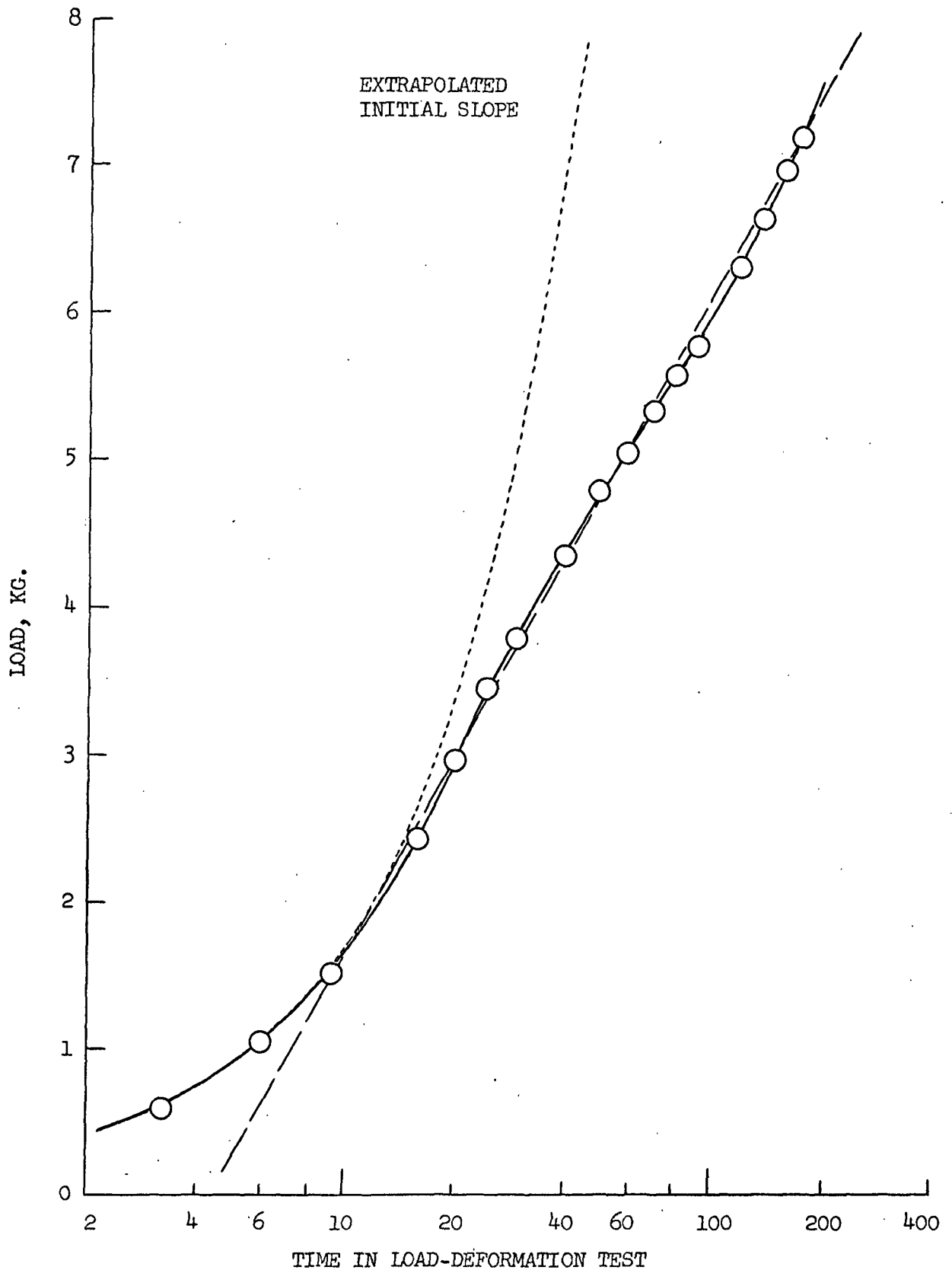


Fig. 9. Load Versus Log Time Plot for Load-Deformation Test of Handsheet 23

load-deformation tests of other specimens, which precludes the use of this semilogarithmic relationship in general analysis of these data. The upper dotted curve represents the result of a calculation of what the load-time plot would be if creep were absent and the load-deformation relationship were linear. Values of dP/dt were calculated from the straight line of Figure 9, and are summarized in Table IV along with time, deformation, and load data taken from the load-deformation curve.

Creep rates can be calculated at various loads and deformations from the slopes of the tangents to the first-creep curves of Handsheet 23. These rates in the appropriate range are given in Table V. The data at the creep load of 7.00 kg. are calculated from the master creep curve of Figure 5 and the time-shift load relationship of Figure 6. One now has a measure of creep rates at various loads and deformations in creep and load-deformation tests. A test of the postulated "equation of state" for first tests of this handsheet can be made by comparing loads at the same creep rate and deformation in both tests. In Figure 10, the logarithm of the creep rate is plotted versus deformation for both types of tests. The intersections of the creep rate versus deformation curves define particular values of creep rate, deformation, and load in creep tests, and the same creep rate and deformation in the load-deformation test. The load in the load-deformation test is obtained directly from the load-deformation curve, since the deformation is known. A comparison of the loads in both tests is given in Table VI. The loads agree well within the accuracy of these data. This analysis, therefore, indicates that the creep rates in continuous first tests of these handsheets may be unique functions of the existing load and deformation and may be independent of the path followed in arriving at those values. The paths differ

TABLE IV
RESULTS OF LOAD-DEFORMATION TEST
OF SPECIMEN 23-5

Relative Humidity, 50%
Temperature, 73°F.
Constant Rate of Deformation, 0.0167 %/sec.

Total Deformation, %	Load, kg.	Time, sec.	(dP/dt) kg./sec. x 10	Creep Rate %/sec. x 100
0.06	0.59	3.6	1.67	0.0
0.10	1.04	6.0	- -	- -
0.16	1.50	9.3	- -	- -
0.25	2.33	15.0	1.31	0.36
0.33	2.95	20.0	0.98	0.69
0.42	3.43	25.0	0.78	0.89
0.50	3.77	30.0	0.66	1.01
0.67	4.32	40.0	0.49	1.18
0.83	4.77	50.0	0.39	1.28
1.00	5.05	60.0	0.33	1.34
1.17	5.31	70.0	0.28	1.39
1.33	5.55	80.0	0.25	1.42
1.50	5.76	90.0	0.22	1.45
1.67	5.94	100.0	0.20	1.47
2.00	6.30	120.0	0.16	1.51
2.33	6.62	140.0	0.14	1.53
2.67	6.93	160.0	0.12	1.55
3.00*	7.22	180.0	0.11	1.56

* Obtained by extrapolation

TABLE V

CREEP RATE VERSUS TOTAL DEFORMATION
IN CREEP TESTS OF HANDSHEET 23

Relative Humidity, 50%
Temperature, 73°F.

Creep Load, kg.	Time in Creep Test, sec.	Total Deformation, %	Creep Rate, %/sec. x 100
3.50	6	0.45	0.33
	10	0.46	0.22
	20	0.47	0.127
4.50	10	0.77	0.63
	20	0.83	0.36
	40	0.88	0.21
	100	0.97	0.098
5.50	10	1.22	1.40
	20	1.32	0.74
	40	1.43	0.39
	100	1.58	0.16
	400	1.80	0.043
6.00	10	1.66	1.87
	20	1.79	0.93
	40	1.92	0.47
	100	2.09	0.19
7.00 ^a	10	2.65	2.18
	100	3.16	0.218

^a The values at 7.00 kg. are calculated from the master creep curve of Handsheet 23.

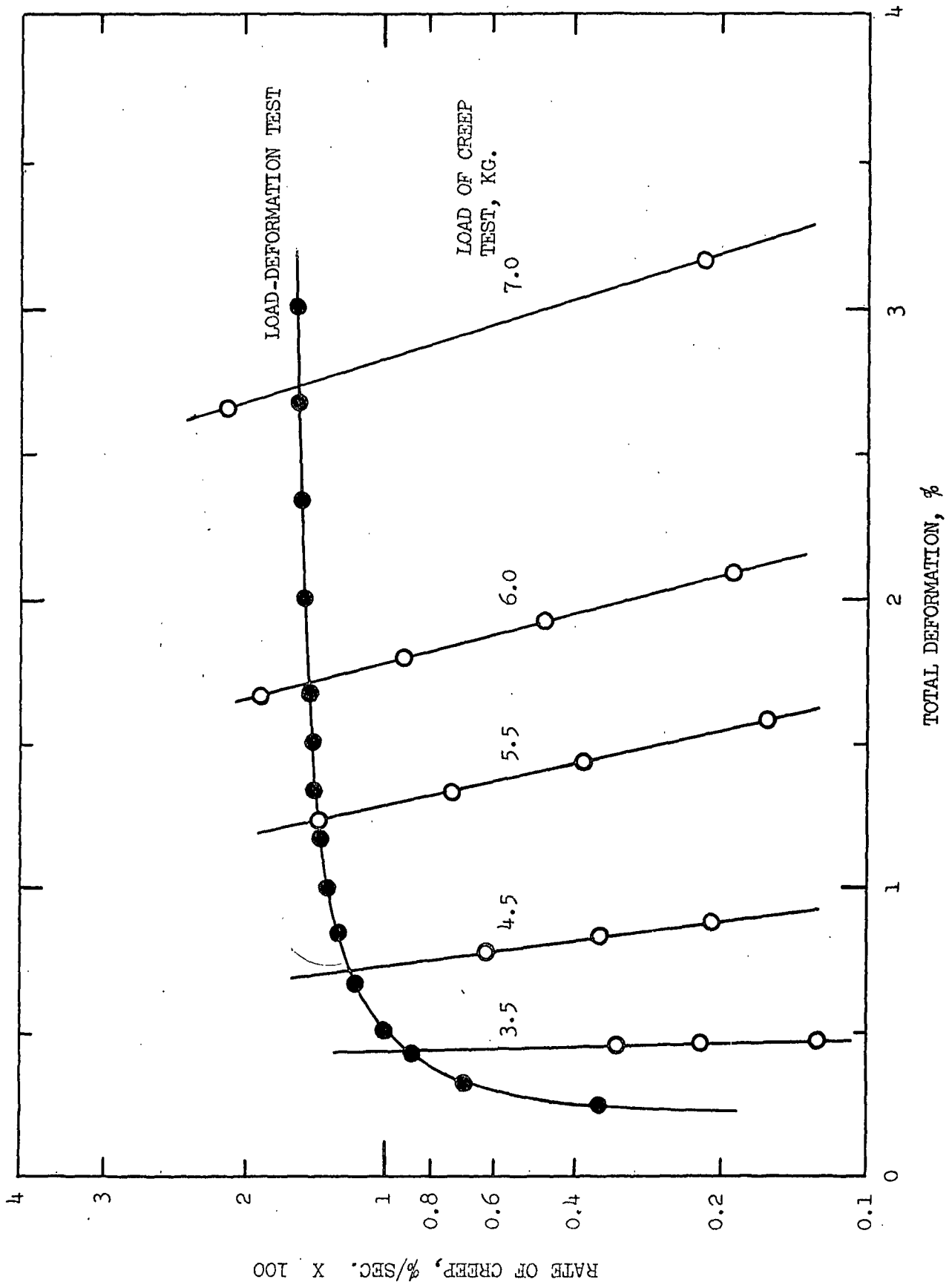


Fig. 10. Creep Rate Versus Total Deformation in Creep and Load-Deformation Tests of Handsheet 23

TABLE VI
COMPARISON OF CREEP RATE-LOAD-DEFORMATION DATA
IN FIRST-CREEP AND LOAD-DEFORMATION TESTS
OF HANDSHEET 23

Creep Rate, %/sec. x 100	Deformation, %	Load, kg.	
		Creep Test	Load-Deformation Test
0.90	0.42	3.50	3.45
1.21	0.70	4.50	4.40
1.40	1.22	5.50	5.36
1.47	1.70	6.00	5.95
1.55	2.77	7.00	7.0

considerably at the higher loads. It was estimated that work done in arriving at the point of intersection of 6 kg. load and 1.70% deformation was approximately 20% greater in the creep test.

It may prove advantageous to analyze load-deformation data in terms of creep rate-deformation-load relationships. If an equation of state concept for first tests were generally applicable over wider ranges of creep rate, an analysis of that type might prove to be independent of the arbitrary rate of straining. It is recognized, however, that data of this type covering a wider range in creep rate are necessary to establish the general applicability of the equation of state concept to first tests of alpha-pulp handsheets.

The results of the preceding analysis offer substantial evidence that the same kinds of response occur at the lower loads as at the higher loads, and that the concept of a speeding-up of response with increasing

load, as indicated by the master creep curve, is valid. Hence, the specimen structure at a given deformation and load is the same whether that point was reached in longer times at lower loads or rapidly at higher loads. It seems most reasonable that the mechanisms of deformation which are rate-controlling are molecular mechanisms of response. Any concept such as macroscopic slipping of fibers, which involves internal damage as a rate-controlling mechanism of deformation would require fortuitous interrelationships of time and load in the different tests to exhibit this behavior. Internal damage undoubtedly occurs in the first deformation of paper. This is a common phenomenon in the straining of many polymers; however, it would not appear to be an important mechanism in determining the rate of creep deformation.

The equation of state concept is of value in qualitatively predicting prerupture response under complex loading patterns. For example, if the load in a creep test were suddenly increased to a higher value, the creep curve which results at the higher load must approach the creep curve obtained if the higher load were applied at zero time. However, at early times after applying the load increment, the total deformation may be greater than would exist at similar short times in a creep test at the higher load. It is at least certain that if the higher load could be attained in both cases at zero time, the deformation would be higher in the incremental application approximately by the amount of delayed deformation which occurred at the lower load. From this, and since delayed deformation is distributed in time, some period of time will be required for the two creep curves to become comparable in shape at equivalent times as predicted by an equation of state. The deformation should be greater,

therefore, in the incremental application test over measurable periods of time. The rate of creep should be lower and the creep curve obtained after the load increment was applied should be flatter at early times and approach the normal first-creep curve at that load as time progressed. Thus, any differences in application of creep loads in creep tests should have a negligible effect on the creep curves at longer times.

An increase in rate of deformation in load-deformation tests results in an increase in load at comparable deformations in order to provide for higher creep rates. In Handsheet 23, the increase in creep rate with load is nonlinear as shown by the family of curves of Figure 10. A slight increase in load causes large increases in creep rate, hence, the load-deformation curves are not expected to vary widely with changes in rate of deformation. This is consistent with the actual experimental behavior of paper at different rates of deformation (42). Changes in load-deformation curves with rate of deformation are much larger for "linear" polymers as indicated by Alfrey (10). Polymers exhibiting linear deformation versus load relationships at various constant times in creep tests would require larger increases in load to maintain higher rates of creep consistent with higher rates of deformation in load-deformation tests. The small change in the load-deformation curves of paper with rate of deformation is a consequence of the nonlinearity of response to load.

The derivation of an empirical equation relating creep rate to load and deformation, based on the unique dependence of rate on those two variables, has not been attempted. It must be pointed out that the derivation of creep parameters from the load-deformation curve poses a complex problem despite the apparent simplicity of these relationships.

It is felt, however, that the "equation of state" principle applied to first-creep and first load-deformation tests may ultimately lead to an analysis of load-deformation data in terms of creep properties.

CREEP RECOVERY PROPERTIES OF ALPHA PULP HANDSHEETS

Creep deformation which is recoverable within reasonable periods of time after removal of the creep load is attributed generally to retarded elastic mechanisms of response. Reversible changes in phase, namely crystallization at the expense of amorphous material, may also account for creep recovery. An interdependence of different mechanisms of response, also can complicate the retarded elastic behavior so that all of the retarded elastic response in the preceding creep test may not be recoverable within reasonable periods of time. In any case, the measured creep recovery is accepted as a means of dividing the preceding creep deformation into general classifications of primary (recoverable) and secondary (nonrecoverable) creep. In order to gain a better understanding of first-creep response, it is necessary to evaluate that response in terms of its recoverability at the test conditions of temperature and relative humidity. It is the purpose of this portion of the investigation to determine the creep-recovery behavior of alpha pulp handsheets at 50% R.H. and 73°F., primarily to characterize the response of first-creep tests to provide a basis for the treatment of creep recovery data in future work. Following an investigation of the effect of residual load on creep recovery, the effect of time of loading and load in first-creep tests on first-recovery response was investigated.

CREEP RECOVERY AS A FUNCTION OF RESIDUAL LOAD

Experimentally, it would be advantageous to obtain creep recovery curves with a small residual load on the specimen. Mathematically, the load of a creep recovery test is considered as a negative quantity equal in magnitude to the load which was removed from the specimen at the start of the test. It is customary, however, to treat creep recovery data without the negative sign except in mathematical equations where both positive creep deformations and negative recovery deformations are considered. If creep recovery response were linearly related to creep recovery load, the creep recovery with a residual load on the specimen would be diminished proportionately by the ratio of residual load to creep load. The load in the second-creep test would be considered equal to the increment of applied load and not to the total load on the specimen, except when creep in the second test progressed beyond the point of the total recoverable response of the creep recovery test. In the latter case, the effective load might be either equivalent to the total load or inadequately defined. Although these observations seem rather obvious, they were checked experimentally for paper to develop a basis for treating creep recovery data.

First-creep tests were run on three specimens of Handsheet 24 at 50% R.H., 73°F., and 3.5 kg. load for a period of 183,000 seconds. Creep recovery curves were obtained at residual loads of 0, 0.30, and 0.60 kg. over a period of about 1,470,000 seconds. Second-creep tests were run on these specimens at 3.5 kg. total load for about 595,000 seconds. Second-recovery tests were run with a reversal in residual load on the specimens which had residual loads of 0 and 0.30 kg. in the first-recovery test. A summary of these tests is given in Table VII. Total creep recovery includes the immediate elastic contraction and the delayed recovery. At zero residual

TABLE VII

CREEP RECOVERY VERSUS RESIDUAL LOAD

Relative Humidity, 50%
Temperature, 73°F.

Test number	21	22	23
Specimen number	24-9	24-8	24-7

First-Creep Test

Load, kg.	3.50	3.50	3.50
Initial stress, kg./sq. mm.	3.64	3.61	3.60
Duration of test, sec.	183,000	183,000	183,000
Total first-creep deformation, %	0.952	0.925	0.928
First-creep strain per unit initial stress, sq. mm./kg. x 1000	2.62	2.56	2.58

First-Recovery Test

Residual load, kg.	0.00	0.30	0.60
Recovery stress, kg./sq. mm.	3.64	3.30	2.98
Duration of test, sec.	1,447,000	1,448,000	1,445,000
Total first-recovery deformation, %	0.624	0.545	0.466
First-recovery strain per unit recovery stress, sq. mm./kg. x 1000	1.71	1.65	1.56

Second-Creep Test

Total load, kg.	3.50	3.50	3.50
Increment of applied stress, kg./sq. mm.	3.64	3.30	2.98
Duration of test, sec.	592,700	592,100	595,000
Total second-creep deformation, %	0.735	0.643	0.577
Second-creep strain per unit applied stress, sq. mm./kg. x 1000	2.02	1.95	1.94

Second-Recovery Test

Residual load, kg.	0.30	0.00	0.60
Recovery stress, kg./sq. mm.	3.32	3.61	2.98
Duration of test, sec.	1,000,000	1,000,000	1,000,000
Total second-recovery deformation, %	0.521	0.588	0.436
Second-recovery strain per unit recovery stress, sq. mm./kg. x 1000	1.57	1.63	1.46

load, the reported recovery deformations include the small deformation due to the 300-gram load which is applied to the specimen in taking the readings. All values of deformation are measured from the length of the specimen just prior to the start of each test and are given in percentage of the initial specimen length. Similarly, the values of stress are calculated by dividing the appropriate value of load by the calculated initial solid cross sectional area A_0 of each specimen. A part of the difference between creep recovery deformations at each residual load are the result of the immediate elastic deformations due to those loads. A comparison of relative recovery was made by calculating the total recovery strain per unit recovery stress.

These data indicate that residual loads in the order of 0.30 kg. will have a significant effect on both the recovery response and the subsequent creep response. Differences in total recovery strain per unit recovery stress indicate a slight nonlinearity in the total recovery deformation to recovery stress relationship. The total recovery deformation was essentially complete at completion of the first-recovery test at all residual loads (see Appendix, Table A), hence, this nonlinearity is not attributable to incomplete recovery. It is noted at all times in the recovery test. The response in second-creep tests was proportional most nearly to the applied increment of stress despite the fact that some of the response in the second-creep test exceeded the total recovery of the preceding recovery test. It must be concluded as a result of these tests that creep recovery curves should be obtained at no load if easiest interpretation of the results is desired. The major difficulty in using a residual load is the inability to compare the deformations of successive creep and recovery tests

on an equivalent basis. As a result of this investigation, the bulk of the creep recovery curves in later work were obtained at no load.

CREEP RECOVERY VERSUS DURATION OF FIRST-CREEP TEST

It was mentioned earlier that the recoverable creep deformation may be used only as a first approximation of the deformation due to configurational elasticity, since deformation due to other mechanisms may also be recoverable and some configurational elastic deformation may not be recoverable at the test conditions. The time distribution of the configurational elastic response cannot be predicted; however, measurements of recovery may be made following first-creep tests of varying duration, and such data might be analyzed together with the shape of the first-creep curve to characterize creep in terms of recovery. The following tests were run to obtain data on recovery versus time of loading in first-creep tests at constant load. Essentially, such data provide the measurement of the recoverability of successive increments of first-creep deformation.

Four specimens of Handsheet 21 were loaded in first-creep for 200, 2000, 30,000, and 587,000 seconds at 3.50 kg. The tests were run at standard conditions of temperature and relative humidity with customary initial specimen dimensions. First-recovery curves were obtained at a residual load of 0.30 kg. A summary of the results of these tests is given in Table VIII. The immediate elastic deformation per unit load was estimated at 0.1%/kg., but apparently may be lower in recovery than in creep. Since recovery continued over very extended intervals of time, it was necessary to make slight extrapolations of the first-recovery curves to zero slope at long times in order to obtain a measure of the total possible recovery

TABLE VIII

FIRST-CREEP AND FIRST-RECOVERY TESTS OF HANDSHEET 21

Relative Humidity, 50%
Temperature, 73°F.

Test number	14	13	11	12
Specimen number	21-3	21-2	21-7	21-6

First-Creep Tests

Creep load, 3.50 kg.

Initial stress, 3.64 kg./sq. mm.

Duration of creep test, sec.	200	2000	30,000	587,000
Total first-creep deformation, %	0.516	0.658	0.916	1.22
Total first-creep strain per unit initial stress, sq. mm./kg. x 1000	1.42	1.81	2.52	3.35

First-Recovery Tests

Residual load, 0.30 kg.

Recovery stress, 3.33 kg./sq. mm.

Total first-recovery deformation at various ratios of recovery time to creep time, %

1:100	--	0.376	0.394	0.400
1:10	0.386	0.419	0.446	0.459
1:1	0.410	0.452	0.509	0.533
10:1	0.421	0.474	0.555	--
Extrapolated total first-recovery deformation, %	0.430	0.500	0.578	0.560
Extrapolated total first-recovery strain per unit recovery stress, sq. mm./kg. x 1000	1.29	1.50	1.74	1.68

in each test. The total extrapolated recovery deformation was greatest in the 30,000-second creep test. Creep in this test continued slightly into the logarithmic creep range, which began at a total first-creep deformation of about 0.85% and at a creep time of about 15,000 seconds (see Figure 13). Further first-creep deformation did not result in increased recovery, but actually, may have caused a decrease in recovery. It is possible, of course, that this decline was due to a variation between specimens. O'Shaughnessy (58) found a maximum in recovery versus time of loading in the creep of nylon. He was unwilling to attribute the slightly reduced recovery at the greater creep test durations to any effect other than insufficient recovery time. The later work on mechanical conditioning in this study indicated that slight reductions in recovery may be expected with increased time of loading. The important effect demonstrated by these data is that a limiting value of total recovery deformation is observed with time of loading in first-creep tests which is reached approximately at the onset of logarithmic creep in first-creep tests.

The recovery curves, shown in Figure 11, differ widely in curve shape with time of loading. At short times of loading, the recovery curve is concave downward. At intermediate times of loading, the recovery curves are sigmoidal, whereas at the longest time of loading, the recovery curve is sigmoidal, but concave upward over most of the experimental time interval. It is presumed that all recovery curves are sigmoidal with a point of inflection at very early times in short-duration creep tests. These differences in recovery curve shape are predictable qualitatively by the Boltzmann superposition principle. In effect, this principle states that the observed recovery curve shall be the result of a hypothetically

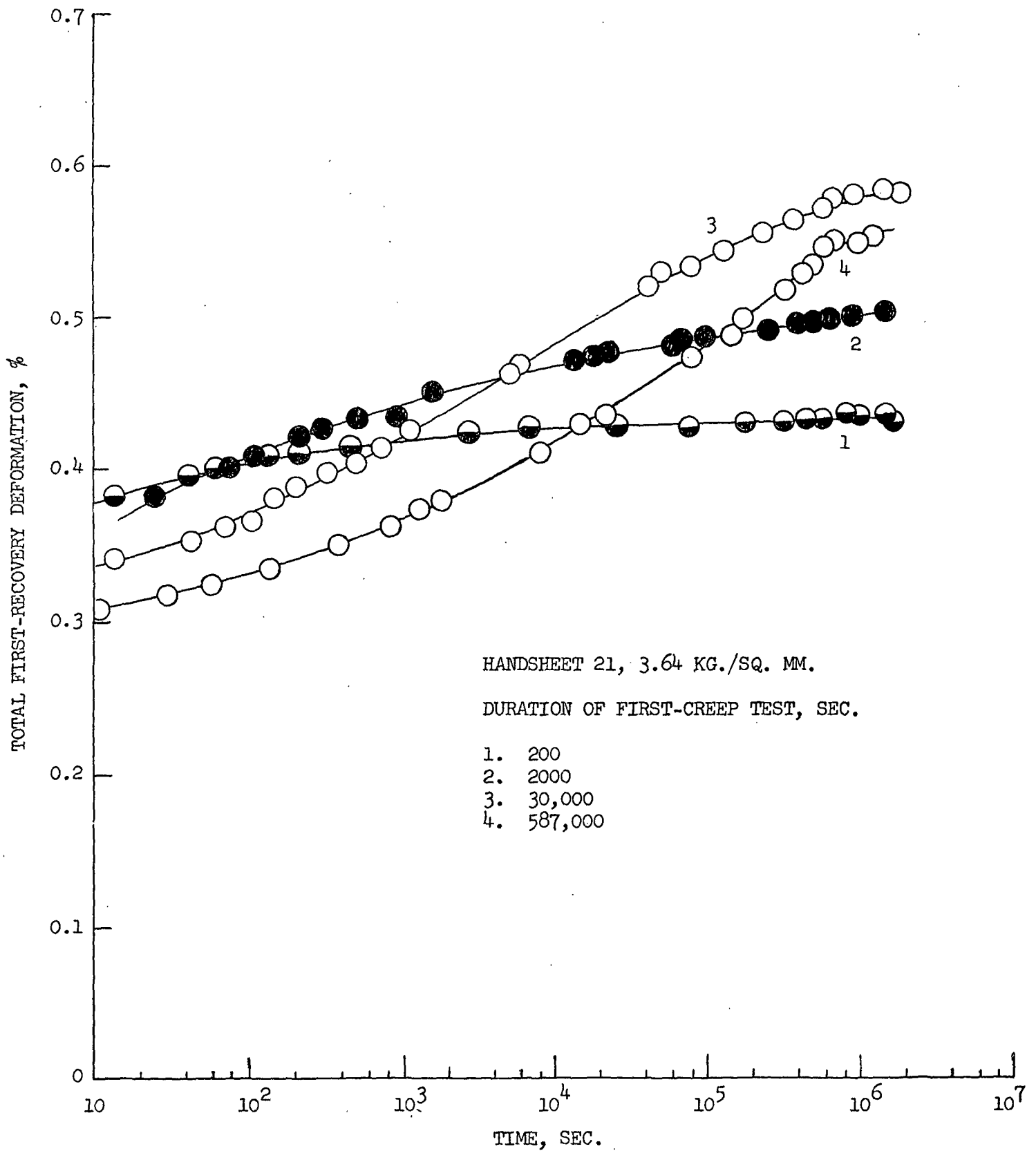


Fig. 11. First-Recovery Curves Following First-Creep Tests of Varying Duration

ideal recovery curve (negative deformation) superposed on the positive delayed elastic deformation which would have occurred in the first-creep test if it were continued. The hypothetical recovery curve is assumed to be identical to the creep curve of configurational elasticity only, which would be obtained on a completely relaxed specimen. Hence, if the creep test is discontinued at a point in time where the creep rate is high, there will be a substantial effect due to superposition and the two superposed deformations will result in a recovery curve resembling that following the 200-second creep test. On the other hand, if the duration of the creep test is increased, this effect will diminish until eventually, when all of the retarded elasticity is accounted for in the creep test, the recovery curve will approach the ideal curve. In these tests, the recovery curve of the 587,000-second creep test would approach the limiting curve shape. The foregoing discussion is limited to ideal polymers which obey the superposition principle. It is not expected to apply quantitatively to paper. It is clear, however, that an analysis of recovery properties will be easiest if long-duration tests are employed.

CREEP RECOVERY VERSUS INITIAL CREEP STRESS

In the preceding section, it was shown that the total first-recovery deformation reached a limiting value with increased time of loading in first-creep tests at the same initial stress, and that in extremely long-duration tests the first-recovery deformation may possibly fall below the limiting value. The following set of tests were run on another hand-sheet at several different loads to determine the effect of initial stress on this recovery behavior.

First-creep tests of varying duration were run on seven specimens of Handsheet 42 at three different loads. Recovery curves were obtained at no load and extrapolated to zero slope at long times. A summary of these tests is given in Table IX. In Figure 12, the total extrapolated first-recovery strain per unit initial stress is plotted versus the total first-creep strain per unit initial stress. It is assumed that the point of immediate application and removal of load will fall on the dashed line of Figure 12, which represents 100% recovery. The experimental curve of Figure 12 will begin at a point on this line if one assumes that the immediate elastic deformation is independent of initial stress. The position of the point on this line is given by the reciprocal of the elastic modulus. For Handsheet 42, the elastic modulus estimated from low-load static tests was 1100 ± 50 kg./sq. mm. The immediate elastic strain per unit initial stress, therefore, will approximate 0.00091 sq. mm./kg.

The first delayed deformation will be due to exponential creep. It will be followed by transitional creep and finally by logarithmic creep. Logarithmic creep began at first-creep strains per unit initial stress of 2.5 to 3.0 sq. mm./kg. $\times 1000$. It should be noted in Figure 12 that the points obtained at all initial stresses and times of loading describe a single curve. There are not sufficient data to establish the shape of the early part of the curve. A linear relationship is shown in this figure; however, it might well be described by a curve. Based on Figure 12, the early deformation is about 60% recoverable. In the transitional creep zone, the recoverability of the first-creep deformation is reduced, whereas in the logarithmic creep zone, the bulk of the deformation is nonrecoverable. This behavior suggests that the first-creep response might be divided

TABLE IX

FIRST-CREEP AND FIRST-RECOVERY TESTS OF HANDSHEET 42

Relative Humidity, 50%
Temperature, 73°F.

Test number	118	117	104	116	113	114	115
Specimen number	42-9	42-10	42-4	42-5	42-8	42-7	42-6
Creep load, kg.	3.5	4.5	4.5	5.5	5.5	5.5	5.5
Calculated initial stress, kg./sq. mm.	3.80	4.87	4.90	5.95	5.95	5.95	5.95
Duration of first-creep test, sec.	10,000	10,100	347,000	5	100	1990	20,000
Total first-creep deformation, %	0.65	1.23	1.90	1.03	1.46	1.95	2.37
Total first-creep strain per unit initial stress, sq. mm./kg. x 1000	1.71	2.52	3.88	1.73	2.45	3.28	3.99
Extrapolated total first-recovery deformation, %	0.54	0.83	0.92	0.83	1.06	1.12	1.14
Extrapolated total first-recovery strain per unit initial stress, sq. mm./kg. x 1000	1.42	1.70	1.88	1.39	1.78	1.88	1.91

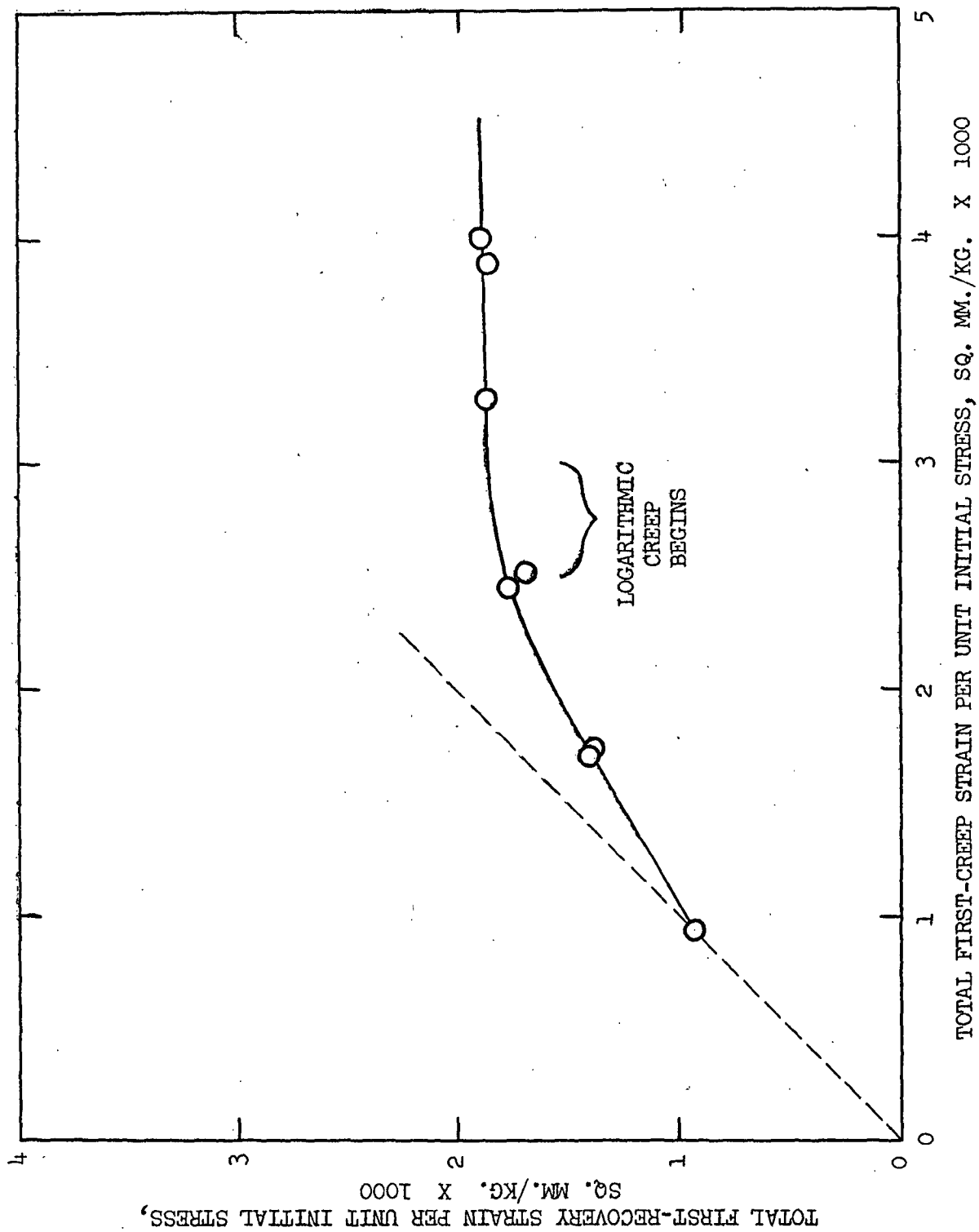


Fig. 12. Relation Between First-Creep and First-Recovery Strains of
Handsheet 42

tentatively into two classes; the partially recoverable deformation which occurs prior to logarithmic creep and the nonrecoverable deformation beyond that point. The partially recoverable deformation includes both exponential and transitional creep, which were defined earlier.

The entire curve of Figure 12 appears to be independent of initial stress. At any combination of initial stress, test duration, and total first-creep deformation prior to the onset of logarithmic creep and the horizontal straight line of Figure 12, the creep strain per unit initial stress is sufficient to establish the recoverability of the deformation. When the limiting value in recovery is reached, however, recovery is fixed by the initial stress and is independent of creep deformation.

In view of the behavior described above, an analysis of the recoverability of successive increments of deformation in the load-deformation test might be very complex. In the load-deformation test, the load increases continuously with time. Hence, with each incremental increase in load some part of the corresponding increase in deformation may be recoverable over the entire load-deformation curve, and no point may ever be reached which corresponds to the onset of logarithmic creep in a creep test. It would appear that the extension of the creep and recovery data described in this section to tests other than creep may not be justified.

The limiting value of total recovery deformation per unit initial stress will be reached at very early times at the higher loads. For example, in the tests at 5.95 kg./sq. mm., the limiting value of total extrapolated first-recovery deformation is essentially reached in the 2000-second test (see Table IX). It would seem that a limiting value of recovery should

also be noted in subsequent tests at this same initial stress. It should be pointed out at this time, however, that in the studies of primary creep which follow, it could not be shown that limiting values in recovery would occur in tests after the first. The fact that limiting values of recovery were not noted in primary creep studies suggests that the observed total first-recovery deformation is not a true measure of the total response due to configurational elastic mechanisms, and that only a portion of the deformation due to that type of mechanism is actually recoverable. The fact that some part of the first-recovery deformation may be due to mechanisms of response other than configurational elasticity further complicates any interpretation of the data just presented. The data are useful primarily in characterizing the various kinds of deformation-time relationships of first-creep tests in terms of their relative recoverability.

MECHANICAL CONDITIONING AND PRIMARY CREEP

It has been shown that substantial portions of the first-creep deformation of paper are not recoverable at the test conditions. The approximate recoverability of the various types of first-creep response were investigated. The first-creep test may be considered as a mechanical conditioning test since the nonrecoverable deformation is not available as a contribution to the deformation in subsequent tests.

Mechanical conditioning is the process of deforming a specimen to some arbitrary degree followed by a period of recovery to remove all or part of the secondary creep so that greater portions of the deformation in subsequent tests will be recoverable. A specimen is said to be mechanically conditioned if the deformation in creep tests after the first is largely

recoverable. In a strict sense, a specimen may be totally mechanically conditioned only if the load and test duration of a subsequent test does not exceed the load and test duration of the mechanical conditioning test. Beyond such limits, the specimen is not mechanically conditioned. It is possible, however, that even within those limits further secondary creep may be noted in subsequent tests. When this behavior occurs, the term "partial mechanical conditioning" will be applied to describe the process by which the specimen was brought to that structural state. The term "partially mechanically conditioned" applied to a specimen has meaning only in comparison with other specimens which exhibit greater percentages of nonrecoverable deformation in similar creep tests. The term is particularly useful to describe the condition of a specimen when the relative humidity is changed during or after creep or recovery tests.

A study of mechanical conditioning is largely a study of primary and secondary creep, which are defined by the relative recoverability of the creep deformation in any given test. These phenomena were studied principally by long-duration cyclic tests of creep and recovery at constant creep load.

SECOND-CREEP RESPONSE AS A FUNCTION OF THE FIRST-CREEP TEST

Paper is frequently subjected to loads and suffers deformations during the processes of its manufacture, and may be received for testing with different mechanical properties due to differences in its previous mechanical history. An interpretation of the creep properties of specimens of undefined mechanical history, therefore, can only be relative. The

relation between the results of first and second-creep tests is basic to an understanding of the mechanical conditioning concept, and may help interpret the creep behavior of specimens which have a poorly defined mechanical history.

Second-creep tests were run on four specimens of Handsheet 21 following first-creep tests of different duration and extended periods of recovery at a residual load of 300 grams. The second-creep tests were run at the same total load used in the first-creep tests, but the test durations were extended beyond the durations of the first-creep tests. A summary of the first-creep and first-recovery tests was given earlier in Table VIII. Detailed data (Tests 11, 12, 13, and 14) are given in Table A of the Appendix. The first and second-creep curves are shown in Figure 13. In both the first and second tests, the total creep deformation represents the increase in length measured from the actual specimen length at the start of each test. The values are reported, however, as percentages of the initial 10.00-inch specimen length.

It may be noted in Figure 13 that the differences in shape between the first and second-creep curves were more pronounced as the duration of the first-creep test increased. It is not possible by simple observation of the shape of a single creep curve to determine whether the specimen has been subjected to previous mechanical tests. A comparison of the deformations in the first and second-creep tests is given in Table X. Different amounts of nonrecoverable deformation occurred in the first-creep tests. In each case, however, the nonrecoverable deformation was approximately equal to the difference in total creep deformation in the first and second-creep

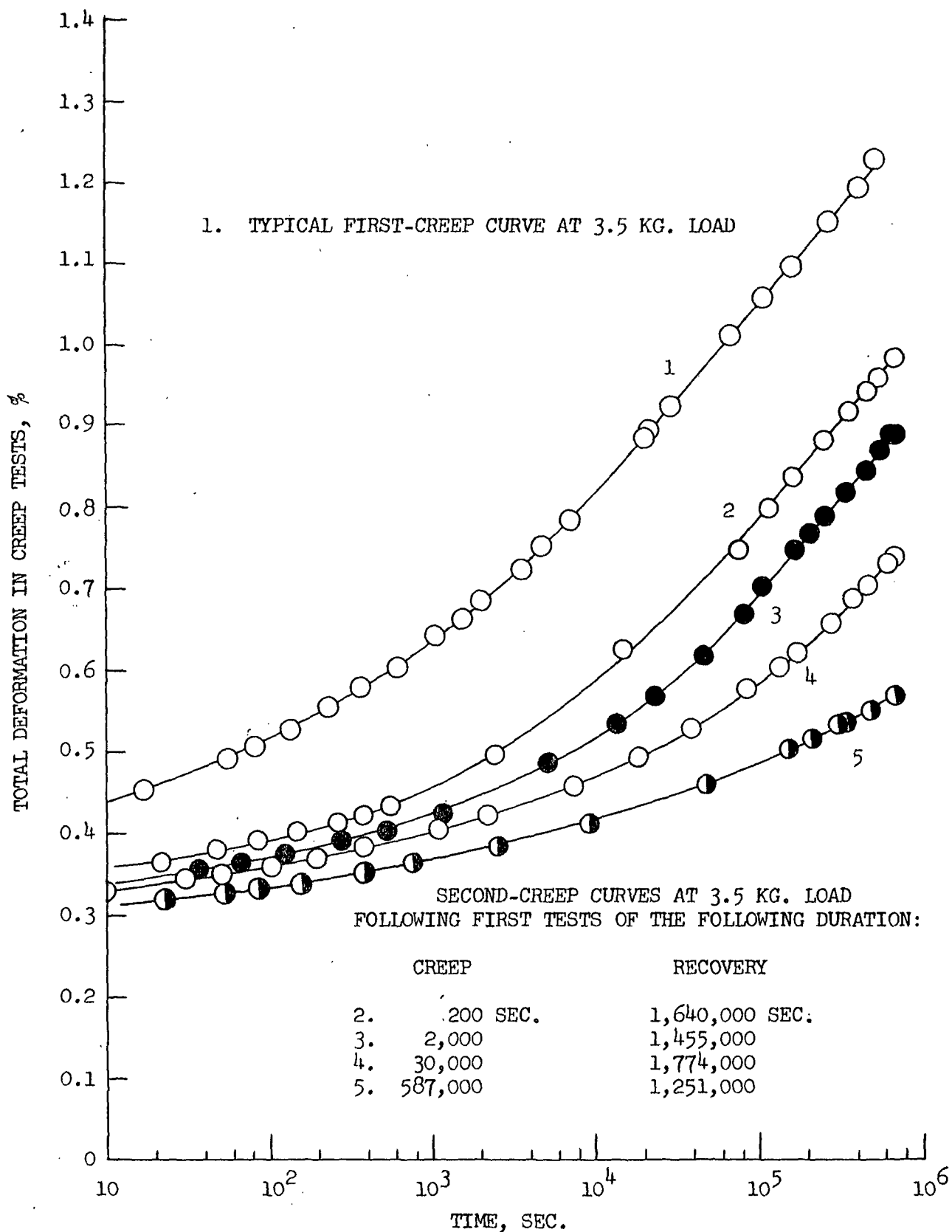


Fig. 13. Second-Creep Curves Following First-Creep Tests of Varying Duration for Handsheet 21

TABLE X
RELATION BETWEEN FIRST AND SECOND-CREEP TESTS

Test number	14	13	11	12
Duration of first-creep test, sec.	200	2000	30,000	587,000
Total first-creep deformation, %	0.516	0.658	0.916	1.221
Total second-creep deformation at time equal to first-test duration, %	0.438	0.473	0.543	0.581
Difference between first-creep and second-creep deformations at time equal to first-test duration, %	0.078	0.185	0.373	0.640
Nonrecoverable deformation in first-creep test, %	0.076	0.148	0.338	0.661

tests at a time equal to the duration of the first-creep test. Stated in other words, the maximum specimen length reached in the first test was reached in the second test at approximately the time of the first test.

A creep rate-time analysis may be useful in revealing differences in the previous mechanical history of different specimens that could not be seen in a visual comparison of the creep curves. It will be recalled that the first-creep curves of Handsheet 23 consisted of exponential creep at early times, which was followed by a transitional zone of creep and logarithmic creep; and that at appropriate loads, all three types of response could be observed in a single creep curve. A plot of the logarithm of creep rate versus the logarithm of time is a straight line of slope equal to $(a - 1)$ for exponential creep (see Equation 1), and a second straight line of slope equal to -1 for logarithmic creep (see Equation 2). The two

lines are connected by a smooth curve of declining creep rate in the transitional zone. In tests of mechanically conditioned specimens, the early part of the creep rate versus time curve (log-log plot) may be either linear or slightly curved, and may be connected to the final straight line of the logarithmic creep zone by an S-shaped curve, where the creep is changing to nonmechanically conditioned response. Irregularities in the creep rate versus time curves could occur at any point in time depending on the previous mechanical history. The nature of the creep rate versus time curve, therefore, may aid in analysis of the creep behavior of specimens of undefined mechanical history.

It should be possible to obtain creep curves which are relatively flat at early times and which rise sharply at some later time in the creep test in tests of extensible specimens which have been mechanically conditioned either in creep tests or during processing of the sheet. It is felt that such behavior is reflected to some degree in the creep curves reported by Rance (34), which rose sharply (semilogarithmic plot) and continued with the steeper slope until termination of the test by rupture. Rance attributed this creep behavior to a mechanism of deformation embracing rupture of fiber-fiber bonds and time-dependent frictional effects. The previous mechanical history of Rance's specimens, however, may have been responsible for this particular type of creep curve. It must be pointed out at this time that mechanical conditioning was investigated primarily at constant temperature and relative humidity in this work, whereas much more complex behavior of the same general type may occur if changes in these external variables were made during a series of creep and recovery tests.

LONG-DURATION MULTIPLE-CYCLE TESTS

In long-duration multiple-cycle tests, the phenomena of mechanical conditioning and the nature of the primary creep response may be analyzed in part by comparing the creep and recovery curves in each cycle and between cycles. A test may be classified as long-duration if the creep and recovery curves are not significantly affected over a wide span of time because the specimen behaves mechanically in accordance with Boltzmann's superposition principle. In tests which meet this requirement, the recovery curve may be compared with the preceding creep curve without correction for superpositional effects. Thus, any differences between a creep and recovery curve in any cycle of a long-duration multiple-cycle test can be attributed to effects other than superposition.

In the following multiple-cycle tests, specimens were subjected to a series of alternate, equal periods of creep and recovery. The same load was used in all creep tests and the recovery curves were obtained at no load. A single cycle consisted of 24 hours of creep and 24 hours of creep recovery. A test period of 24 hours was long enough to classify the tests as long duration for most of the loads used in this investigation.

A number of orienting experiments indicated that the effectiveness of mechanical conditioning depended on the creep load and the test duration. The relation between total first-recovery deformation and total first-creep deformation in tests at different loads and of different duration (see Table IX) suggests that the deformation attained in the first-creep test cannot alone serve as an index of the boundary between mechanically conditioned and nonmechanically conditioned response. The following tests

were run to obtain information regarding the mechanical conditioning and primary creep behavior of alpha-pulp handsheets.

TYPICAL MULTIPLE-CYCLE TEST AT 50% R.H.

Most of the tests in this work were multiple-cycle tests of 24-hour duration in creep and in recovery. From a study of all of these tests, it was possible to establish that certain patterns of behavior were typical of all tests. At 50% R.H., Test 39 on Specimen 33-8 was selected for presentation since it was extended to seven cycles.

The specimen was loaded to 3.50 kg. for 24 hours in the first-creep test, which was followed by a 24-hour period of recovery at no load. The specimen was reloaded to 3.50 kg. in a second-creep test for 24 hours followed by another 24-hour no-load recovery period, and so on for 6 cycles. Following the sixth cycle, the recovery period was extended from 86,400 seconds (24 hours) to 346,000 seconds and a seventh cycle of creep and recovery was run.

The results of these tests are summarized in Table XI. Note particularly that four cycles are required before the creep and recovery curves of subsequent cycles became nearly constant. The specimen length increased, however, by about 0.033% in each additional cycle after the third.

The creep curves of the first 6 cycles are shown in Figure 14. The seventh-creep curve (following a longer recovery time) was nearly identical to the sixth and is not shown. The deformation axis of Figure 14 is the total deformation of the specimen relative to its initial length. The actual deformation in any test other than the first, measured from the specimen length prior to the start of each test, will be less than the

TABLE XI
MULTIPLE-CYCLE TEST 39

Relative Humidity, 50%
Temperature, 73°F.
Specimen 33-8
Initial Micrometer Reading, 0.8270 inches
Duration of Each Test, 24 hours or
as indicated

Cycle Number	Total Creep Deformation in 24 hr., %	Total Recovery Deformation in 24 hr., %	Increase in Specimen Length in Each Cycle, %	Total Deformation at End of Each Cycle, %
1	1.007	0.625	0.382	1.007
2	0.685	0.589	0.096	1.103
3	0.628	0.577	0.051	1.154
4	0.600	0.566	0.034	1.188
5	0.600	0.569	0.031	1.219
6	0.595	0.563	0.032	1.251

Total recovery at 346,000 sec., 0.593%

7	0.595	0.559	0.007	1.258
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Total recovery at 2,467,000 sec., 0.627%

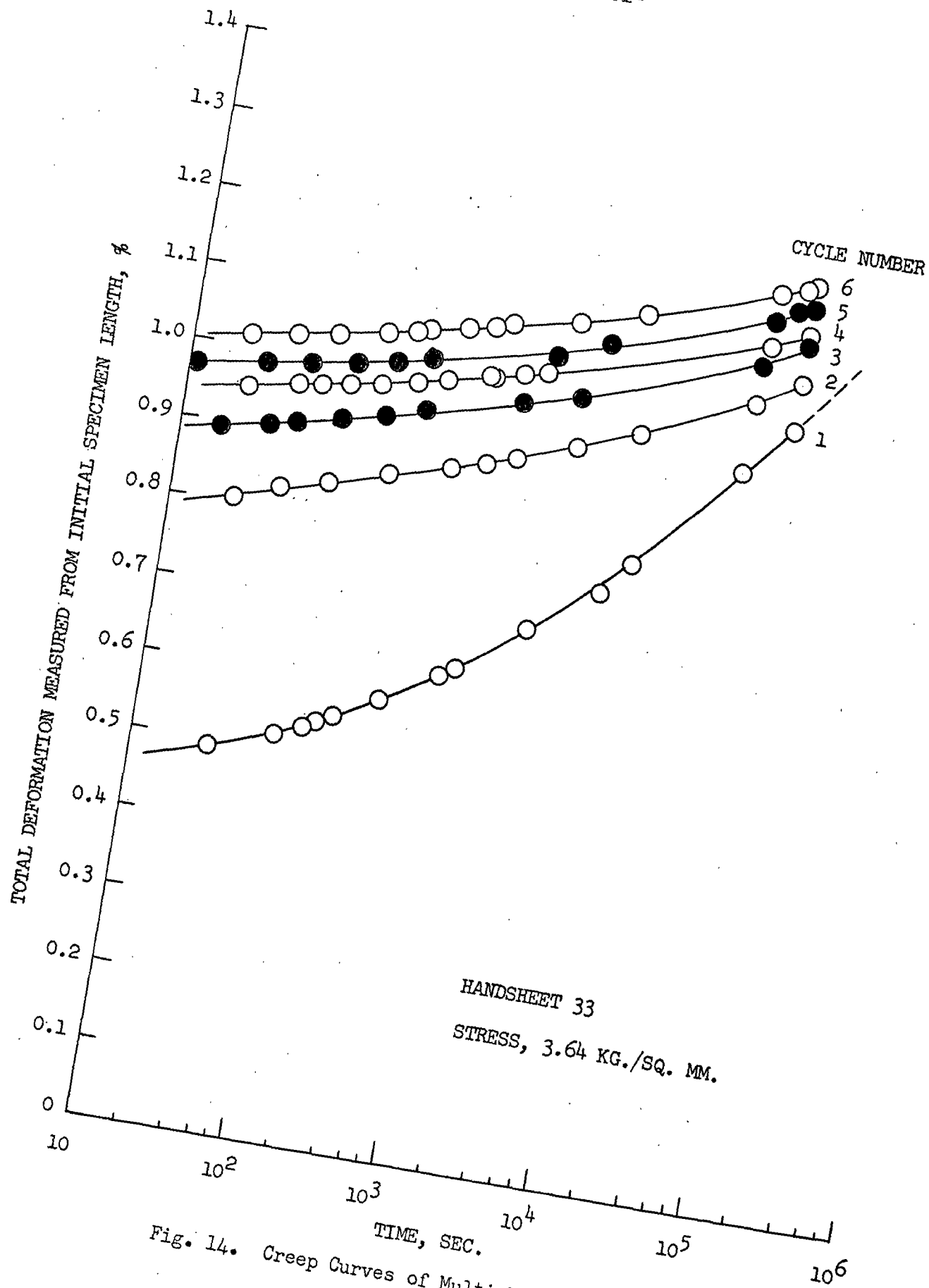


Fig. 14. Creep Curves of Multiple Cycle Test 39

ordinate indicates (see Table XI). The creep tests were plotted in this manner in order to separate the curves and to illustrate the continuing increase in specimen length in each cycle.

By definition, the total primary creep in the second-creep test can equal only the total first-recovery deformation, unless a change in the time-dependency of the recoverable response has occurred. A total second-creep deformation of 0.625% would be expected and an actual deformation of 0.685% was measured. The additional deformation of 0.06% must be attributed to secondary creep effects, since the effect of superposition of continued first-creep and first-recovery tests can be neglected. The increased deformation corresponds closely to the deformation which would have occurred in the first-creep test between 24 and 48 hours. The specimen length increased in each cycle by an amount which approximated the increase which would have occurred in an additional 24-hour increment in the first test. This, however, was not a general effect. It was often observed that the increase in specimen length in each successive creep test did not keep pace with the extrapolated first-creep test at the same total time under load. At least part of the reduction in the creep response in successive cycles must be attributed to a reduction in secondary creep. All of the decrease cannot be due to this source since the recovery response also diminished in the early cycles and the increase in specimen length at the end of the cycle (the end of the recovery test) increased by more than the expected secondary creep. In order to explore the reasons for this effect, one must turn to the recovery curves as a means of assessing the primary creep response of the various creep tests.

The second and sixth-creep curves and the second and sixth-recovery curves are shown in Figure 15. A straight-line extrapolation of the second-creep curve starting at about 1000 seconds would yield a 24-hour deformation of 0.625% which is equivalent to the total 24-hour recovery deformation of the first cycle. The rise in deformation near the end of the second-creep test is not typical of primary creep. It diminishes with continued cycles. It may be noted that the creep deformation between 10 and 1000 seconds is reduced in the first few cycles. In these 24-hour tests, superpositional effects will not significantly affect the creep curve at early times. Also, it is not likely that secondary creep will constitute a significant part of this early deformation in tests after the first. The creep curve at early times, therefore, should approximate the true primary creep curve, which would be obtained after a 24-hour mechanical conditioning test and an extremely long period of recovery. In essence, the creep curves between 10 and 1000 seconds in 24-hour tests after the first should represent primary creep response and the deformations in that range of time should be recoverable within reasonable times in succeeding recovery tests. Primary creep was defined earlier as that deformation which is recoverable within reasonable periods of time after removal of load. The reduction in slope of the creep curves in the first few cycles suggests that an actual decline in primary creep accounts for the decline in recovery which is noted. This becomes more apparent when the total recovery deformation of 0.627% in the seventh-recovery test in almost 29 days is compared to an estimated 0.67% expected in the first-recovery test in a similar period of time.

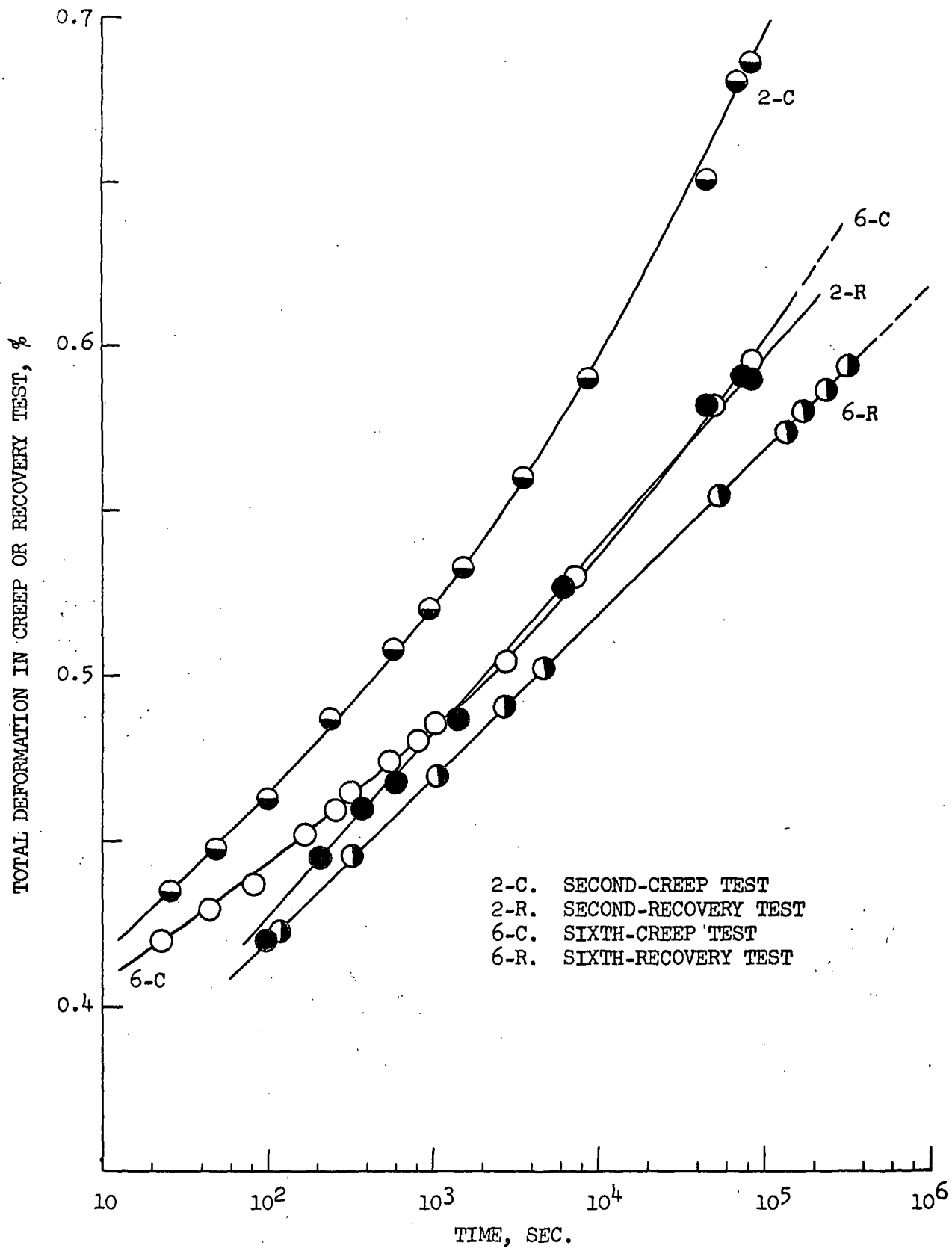


Fig. 15. Creep and Recovery Curves in Second and Sixth Cycles of Multiple-Cycle Test 39

This behavior indicates that recoverability may not be a true measure of the creep deformation due to the mechanisms of the type which account for the recoverable deformation. Hence, the first-recovery deformation may be used as a measure of primary creep in the first-creep test only by definition, and may not indicate a magnitude of first-creep deformation of a particular kind. It is felt that the reduction in primary-creep response in succeeding cycles of multiple-cycle tests is due to the formation of a metastable molecular structure which inhibits the recovery of these deformations.

It may be noted in Figure 15 that the recovery curves fall lower on the deformation axis than the creep curves. This was true for any cycle and is general at all higher stresses in multiple-cycle tests. If the specimen responded in a manner indicating compliance with the superposition principle, the curves should be nearly identical over most of the experimental time interval. The fact that they are not indicates noncompliance with the superposition principle and further suggests the presence of mechanisms of response other than simple configurational elasticity. It is suggested that the mechanisms of response responsible for the reduction in primary creep are of the type which account for the differences between the creep and recovery curves.

It is reasonable to assume that if primary creep were diminished because of additional structural changes accompanying truly permanent deformations, a similar effect should become apparent in comparing recovery curves of multiple-cycle tests with recovery curves following first-creep tests of different times of loading. The creep recovery curves of the

first and sixth cycles of Test 39 are compared in Figure 16 with the first-recovery curves following first-creep tests of 72 and 240-hour duration. These latter curves are taken from Tests 38 and 36 on Specimens 32-8 and 30-11, respectively, which have comparable creep properties to Specimen 33-8 of Test 39. The total recoverable deformations of these tests are in the same order of magnitude, since the limiting value of recoverable deformation was essentially reached in 24-hour creep tests. An increase in continuous time under load reduced the recovery deformation at early times, but the recovery curves are steeper at longer times on the semilogarithmic plot. An increase in intermittent time under load merely reduced the level and the slope of the recovery curve as illustrated by Curves 1 and 4 of Figure 16, without changing its shape. Apparently, the response of longer retardation times in the creep test changes the distribution in time of the delayed recovery deformation by reducing the recovery at early times and allowing this recovery to occur later in time. Longer intermittent time under load reduces the recovery deformation almost proportionately over the entire recovery period. The differences between the primary creep and recovery curves would be greater, therefore, in longer-duration tests. Conversely, it might be assumed that these differences would be diminished in shorter-duration creep tests, although this cannot be demonstrated effectively. It is clear, however, that the disagreement between the long-duration creep and recovery curves must be attributed to mechanisms of deformation of longer retardation time. This subject is explored further by a comparison of primary creep and recovery curves at different creep stresses.

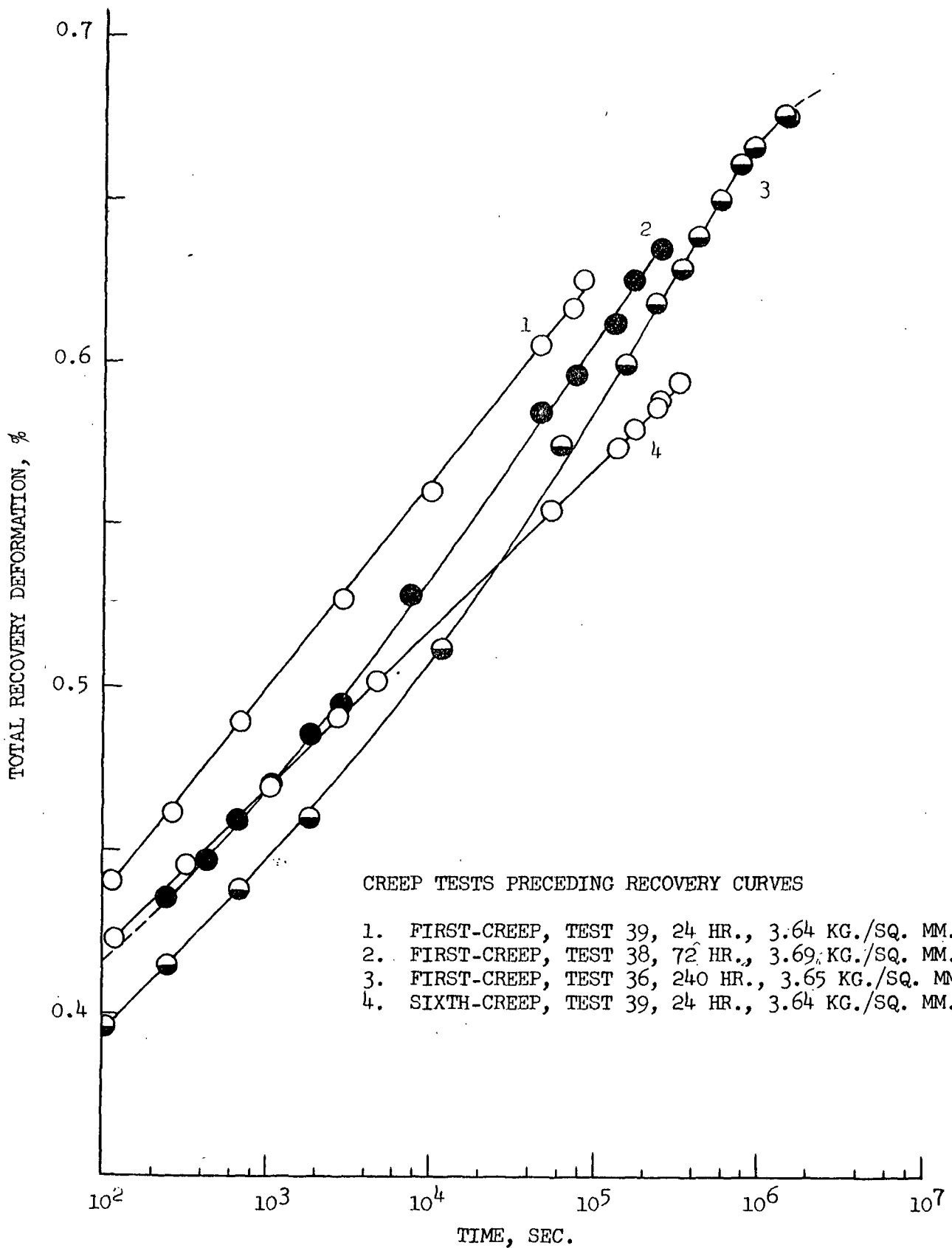


Fig. 16. Comparison of Recovery Curves Following Various Creep Tests

PRIMARY CREEP AS A FUNCTION OF STRESS

The effect of initial stress on primary creep and recovery behavior at 50% R.H. is typified by the following tests of Handsheet 55. It was noted in this study that the following results are typical of behavior in tests at relative humidities below about 65%. The fourth cycles of multiple-cycle tests at three different creep stresses will be considered. The behavior in each of these multiple-cycle tests was analogous to that of Test 39. A summary of the tests is given in Table XII. Note that the nonrecoverable deformation at the start of the fourth cycle is greater at the higher stresses, since the specimens were mechanically conditioned at the stress of the fourth cycle. If all of the tests were run on a specimen following mechanical conditioning tests at 5.45 kg./sq. mm., the response in the fourth cycle would be slightly lower at the two lower stresses, but would not affect the general behavior in any other way. The fourth-creep and fourth-recovery curves (24-hr. tests) are plotted in Figure 17. The effect of stress is two-fold. The disagreement between the creep and recovery curves is greatest at the higher stresses and the shape of the primary creep curve changes with stress. At the lowest stress, the specimen conforms to the superposition principle as indicated by the close agreement between the creep and recovery curves. The creep curve at the lowest stress is concave upward over most of the experimental time interval. At higher stress, the creep curve is almost linear, disregarding the rise in response in the last decade of log time which was shown to be characteristic of nonmechanically conditioned behavior. The change in shape of the creep curve is evidence of a nonlinear relationship between total primary-creep deformation and creep stress at constant test durations.

TABLE XII

PRIMARY CREEP OF HANDSHEET 55
IN MULTIPLE-CYCLE TESTS

Relative Humidity, 50%
Temperature, 73°F.
Duration of Each Test, 86,400 sec.

Test number	78	77	79
First-creep stress, kg./sq. mm.	3.49	4.48	5.45
Total first-creep deformation in 24 hours, %	0.65	1.23	2.03
Nonrecoverable deformation at start of fourth cycle, %	0.27	0.71	1.40
Total fourth-creep deformation at 100 sec., %	0.354	0.508	0.650
Relative delayed deformation between 100 and 10,000 sec., %	0.086	0.126	0.152

These primary creep curves could be combined into a master creep curve for primary creep by reducing the deformation in proportion to stress and shifting the reduced curves along the time axis. The agreement in the regions of overlap is satisfactory; however, the time-shift requirement cannot be measured with any degree of accuracy since small changes in the level of deformation can have a very large effect on the required shift in log time without affecting the fit in the regions of overlap. The construction of master creep curves for primary creep was not employed for the analysis of primary creep data since greater accuracy is clearly needed; however, the changes in curve shape within an experimental time interval

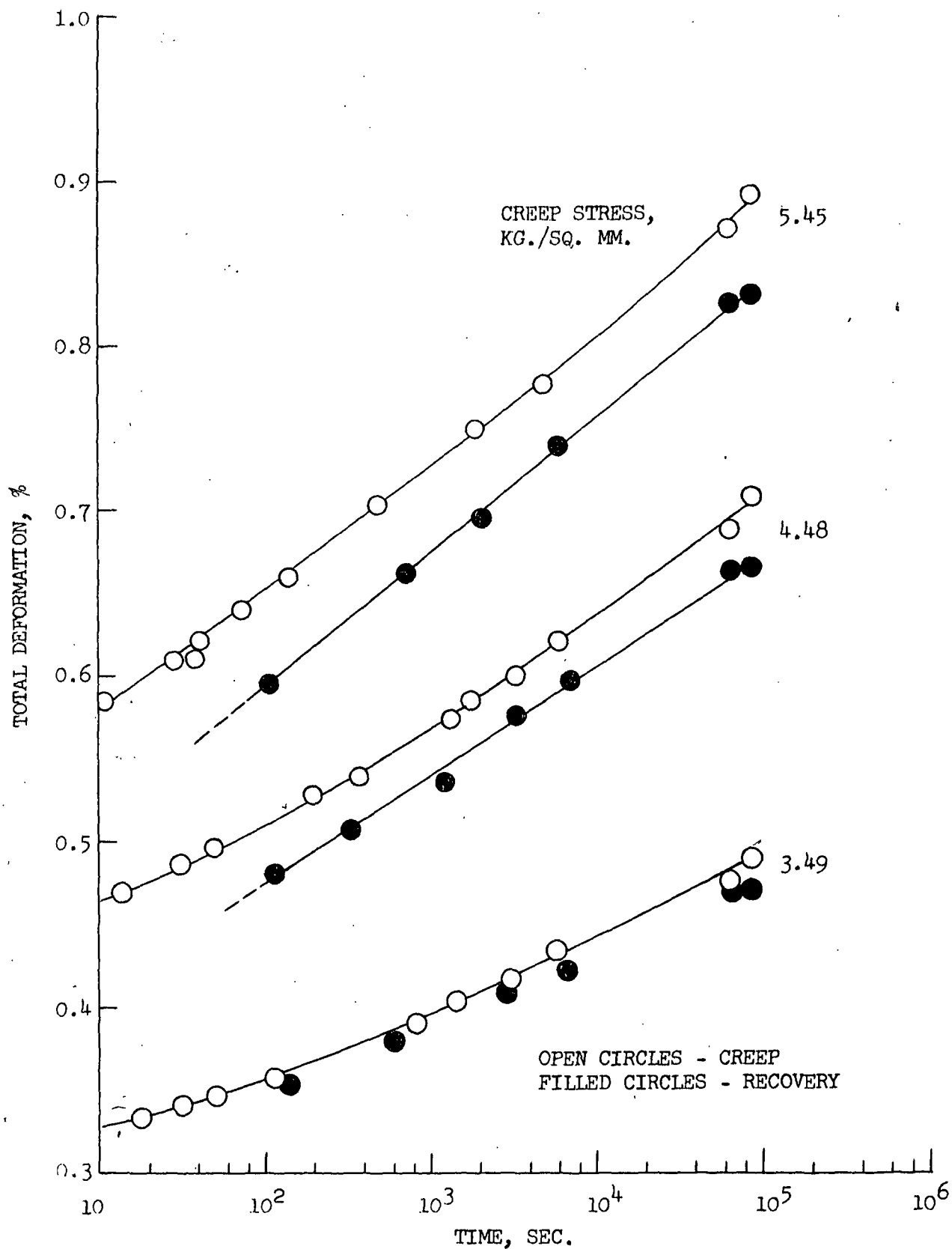


Fig. 17. Primary Creep and Recovery Curves at 50% R.H.

as a function of stress are consistent with the master creep curve concept. The slopes of the logarithmic primary creep curves were approximately proportional to stress and the early response at any load could be described best as exponential or transitional creep. An estimated K/S_0 for logarithmic primary-creep was about 6×10^{-5} sq. mm./kg. compared to about 3.2×10^{-4} sq. mm./kg. for logarithmic first-creep response of these specimens.

There is reason to suspect that primary creep response of the logarithmic type may be responsible for the disagreement between primary creep and recovery curves. This confirms the earlier observation that the response of longer retardation times at constant load influenced the shape of the recovery curve in a manner tending to promote disagreement between the primary creep and recovery curves of long-duration tests. The disagreement is a function of stress partly because logarithmic creep begins at earlier times at the higher stresses and more of that deformation occurs in equal test durations. The disagreement would be enhanced at the lower stresses if longer test durations were employed. Thus, it appears that compliance with the superposition principle might be demonstrated in tests at any stress if one selects test durations which minimize the amount of logarithmic creep. Steenberg's (5) statement that mechanically conditioned paper obeys the superposition principle in short-duration cyclic tests except for early times would appear to be correct except that the disagreement at early times may be due to structural changes which occur at the longer times. If a longer cycle were chosen, the disagreement at early times in each cycle would be greater and vice versa.

Test 40 on Specimen 32-3 at 83% R.H. was selected to illustrate the typical primary creep and recovery behavior which is noted in 24-hour

multiple-cycle tests at the higher relative humidities. A summary of these data is presented in Table XIII and the primary creep and recovery curves are shown in Figure 18. The testing procedures and data at relative humidities other than 50% are summarized in a later section relating creep properties to relative humidity.

The curves at 3.65 kg./sq. mm. represent the fourth cycle of a multiple-cycle test of 24-hour duration in creep and recovery at the same creep stress. The stress was reduced to 2.92 kg./sq. mm. in the fifth cycle, etc. The same general disagreement between creep and recovery curves is noted as at 50% R.H. except that the recovery curves are steeper relative to the creep curves. The primary creep curves at the higher stresses are slightly concave downward. This is usually characteristic of the early response at higher stresses and higher relative humidities. At higher relative humidities, a change in the effect of stress on curve shape was observed relative to the behavior at 50% R.H. This is illustrated in Figure 19. The total creep deformations at 100 seconds and the relative delayed deformations between 100 and 10,000 seconds are plotted versus creep stress. In both cases, the total deformation versus stress relationship is nonlinear. If the master creep curve concept were applicable to the primary creep curves as it is for first-creep curves, a plot of the relative delayed deformation between any two experimental times versus stress should describe a sigmoidal curve which becomes a straight line at higher stresses when the response between the two arbitrary times is logarithmic. This straight line would pass through the origin and represent the proportionality between slope of the logarithmic creep curve and stress. This type of curve is noted for

TABLE XIII

PRIMARY CREEP AT 83% R.H.
IN MULTIPLE CYCLE TESTS

Temperature, 73°F.
Duration of Each Test, 86,400 sec.
Test 40
Specimen 32-3

Cycle number	1	4	5	6	7
Creep stress, kg./sq. mm.	3.65	3.65	2.92	2.08	1.35
Total creep deformation in 24 hr., %	3.02	0.95	0.71	0.49	0.29
Nonrecoverable deformation at start of each cycle, %	0.0	2.23	2.28	2.29	2.30
Total creep deformation in 100 sec., %	- -	0.725	0.530	0.342	0.188
Relative delayed deformation between 100 and 10,000 sec., %	- -	0.139	0.130	0.098	0.054

Handsheet 55. It could be demonstrated in first-creep tests as well for any handsheet where the master creep curve can be constructed in the manner described earlier. At 83% R.H., the curve begins in the same manner but is not stress proportional at the higher creep stresses. The primary creep curves in this range of stress and time tend to become parallel, and the relative delayed deformation increases only slightly with increased stress.

This is the type of behavior reported by Leaderman (3) for nylon at high stresses and for viscose rayon at high relative humidities. The steeper recovery curves and the concave-downward shape of the primary creep curve is common in nylon at higher loads. Leaderman postulated that these effects in nylon were due to crystallization during the creep test,

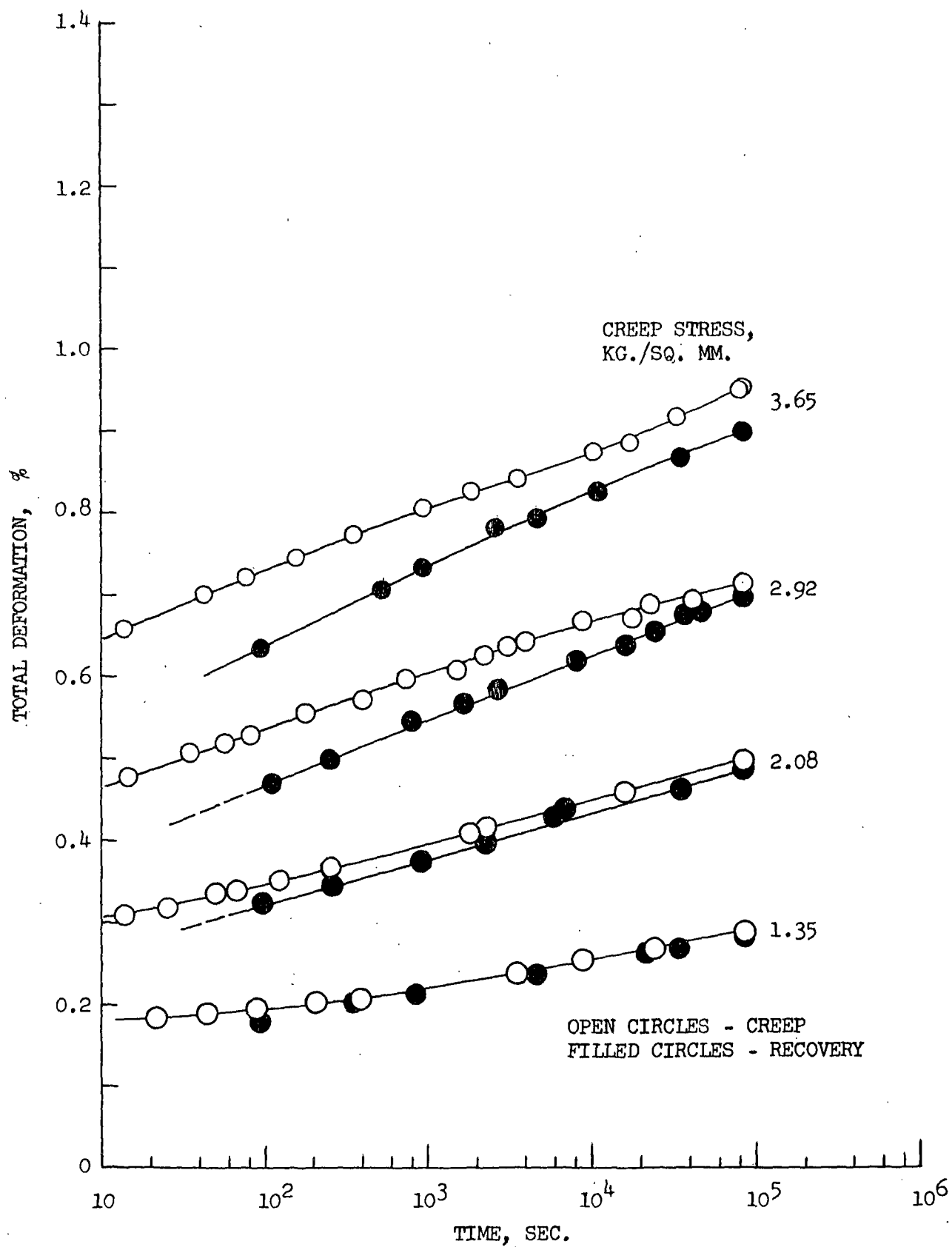


Fig. 18. Primary Creep and Recovery Curves at 83% R.H.

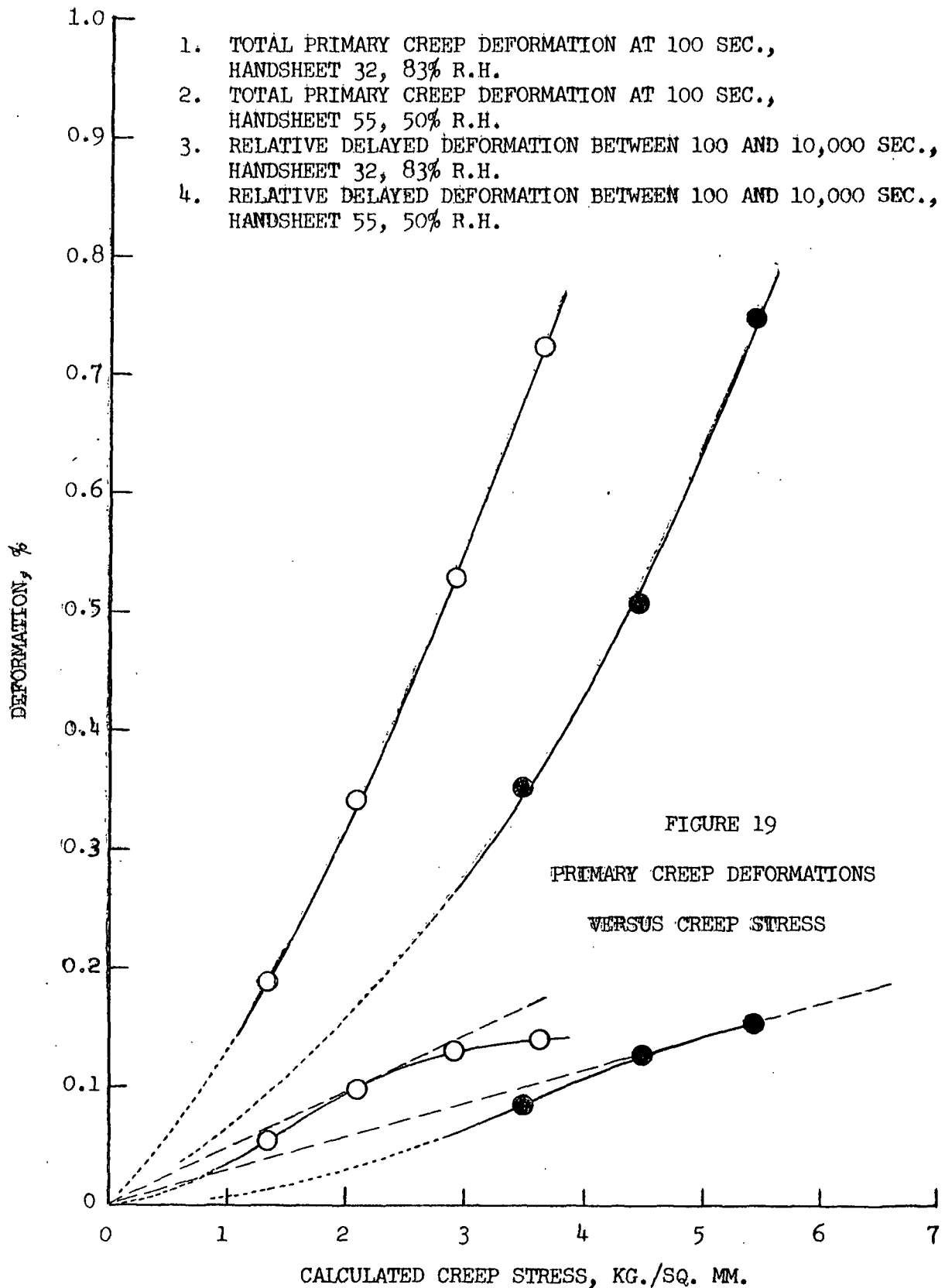


Fig. 19. Primary Creep Deformations Versus Creep Stress

and melting of the stress-induced crystallite growth during the recovery period. Whether Leadermen's hypothesis is correct for nylon has not been confirmed. It is based on the same type of evidence shown in Figures 17 and 18, and could be applied to paper as well. The present writer feels that the role of the crystallite cannot be denied; however, the precise mechanisms are a matter of doubt.

The similarity between the first-creep and primary-creep response at 50% R.H. is evident from the general shapes of the various curves at different stresses. The chief differences lie in the amount of response and in the recoverability of the deformation. It is tempting to consider, therefore, that the mechanisms of response in first-creep tests are of the same type as occur in primary creep. In this sense, the ability to mechanically condition a specimen would involve simply the formation of new molecular structures of varying degrees of relative permanence, ranging from a condition of metastability to irreversible changes in crystalline structure.

DEPENDENCE OF CREEP PROPERTIES ON SHEET STRUCTURE

The mechanical properties of paper may be considered to be functions of sheet structure and fiber property variables. Sheet structure is related principally to the macroscopic dimensions and geometrical arrangement of the fibers in the sheet, whereas fiber properties are related to those inherent characteristics of the fibers which do not depend on the formation of a sheet for their existence.

One has some control in determining the sheet structure principally by variations in beating and wet pressing. Increased beating and wet pressing

may alter the numbers, areas, strengths, and geometrical arrangements of fiber-fiber bonds throughout the sheet. In addition, beating will change the fiber-size distribution to promote more effective packing of the fibrous elements in the sheet. Both beating and wet pressing within normal limits increase the bonded area, the solid fraction, and the tensile strength. It is generally assumed that the extent of interfiber bonding determines the strength of the sheet, although it may not be the sole factor in determining strength in the plane of the sheet. The load at equivalent deformations in load-deformation tests increases with increased beating and wet pressing. Little is known about the mechanisms by which these changes in prerule response come about. In view of the heterogeneous macroscopic structure of paper, it seems inevitable that changes in sheet structure, particularly interfiber bonding, will affect the stress distribution patterns throughout the sheet to a considerable degree. It seems essential, therefore, to investigate the dependence of creep behavior on sheet structure. In the following series of tests, sheet structure was varied principally by changes in the extent of beating and wet pressing.

EXPERIMENTAL PROCEDURES

Most of the tests were multiple-cycle tests of 24-hour duration in creep and recovery. All tests were run at 50% R.H. and 73°F. on specimens in the as-dried condition. The range of creep loads was limited at the upper level by the tensile strength of the specimens in long-duration tests. Generally, at least 3 different loads were used to obtain total 24-hour first-creep deformations from less than 1 to over 2% in tests on each handsheet. All creep testing procedures have been described earlier, and in all cases the recovery curves were obtained at no load.

The handsheets selected for these tests are characterized in Table II. All handsheets with the exception of Handsheet 52 vary only in degree of beating and wet pressing. Handsheet 52 was prepared from the on-20-mesh fraction of 775 cc. S.-R. freeness pulp. The pulp was screened in a Bauer-McNett classifier in accordance with Institute Method 415. About 32% of the pulp was retained on the 20-mesh screen. This fraction consisted of the longer fibers, and possibly contained an increasing percentage of thicker-walled fibers. Fiber fines were discarded. The handsheets were visibly coarse in structure and surface texture.

RESULTS AND DISCUSSION

A summary of the tests in this series is presented in Table XIV. Only 24-hour first-creep and 24-hour first-recovery tests are considered. In a few cases, however, total 24-hour first-creep deformations were estimated by extrapolation of shorter-duration tests. These data are enclosed in parentheses. Data obtained by interpolation of longer-duration tests may be recognized by the absence of recovery data.

In those tests which were extended by additional cycles of creep and recovery, it was noted that the patterns of behavior of all handsheets in multiple-cycle tests were substantially the same as described earlier. Consequently, these additional data were not included.

FIRST-CREEP PROPERTIES

The total first-creep deformation versus initial stress relationships are illustrated conveniently by a log-log plot as shown in Figure 20. The

TABLE XIV

SUMMARY OF FIRST-CREEP AND FIRST-RECOVERY TESTS
ON HANDSHEETS OF DIFFERENT SOLID FRACTIONRelative Humidity, 50%
Temperature, 73°F.

Test No.	Spec. No.	Handsheet Solid Fraction, %	Load, kg.	Initial Stress, kg./sq. mm.	Total 24-Hr. First-Creep Deformation, %	Total 24-Hr. First-Recovery Deformation, %
59	47-6	31.6	2.00	1.95	0.64	0.44
58	47-9		2.80	2.72	1.23	0.66
66	47-4		2.80	2.73	1.27	0.68
50	47-8		3.50	3.39	2.17	0.88
46	47-11		3.50	3.44	2.19	--
60	48-4	40.6	2.80	2.73	0.78	0.52
67	48-5		3.50	3.43	1.36	0.71
61	48-2		3.50	3.43	1.40	0.72
47	48-10		3.50	3.45	1.52	--
68	48-1		4.00	3.92	1.93	0.85
63	50-6	50.6	2.80	2.81	0.73	0.49
62	50-5		3.50	3.53	1.16	0.64
48	51-2		3.50	3.41	1.19	--
64	50-7		4.50	4.53	(2.30)	--
65	52-8	39.9	2.80	2.69	0.59	0.41
69	52-4		3.50	3.30	1.04	0.60
49	52-12		3.50	3.44	1.27	0.64
70	52-7		4.50	4.27	1.82	0.78
20	23-6	47.3	2.00	2.06	0.34	--
18	23-10		3.50	3.60	0.99	--
17	23-9		3.50	3.60	1.02	--
16	23-8		4.50	4.62	1.84	--
15	23-7		4.50	4.63	1.84	--
19	23-13		4.50	4.68	1.84	--
27	23-1		5.50	5.70	2.72	--
25	23-4		6.00	6.20	(3.30)	--
26	23-11		6.00	6.19	(3.37)	--

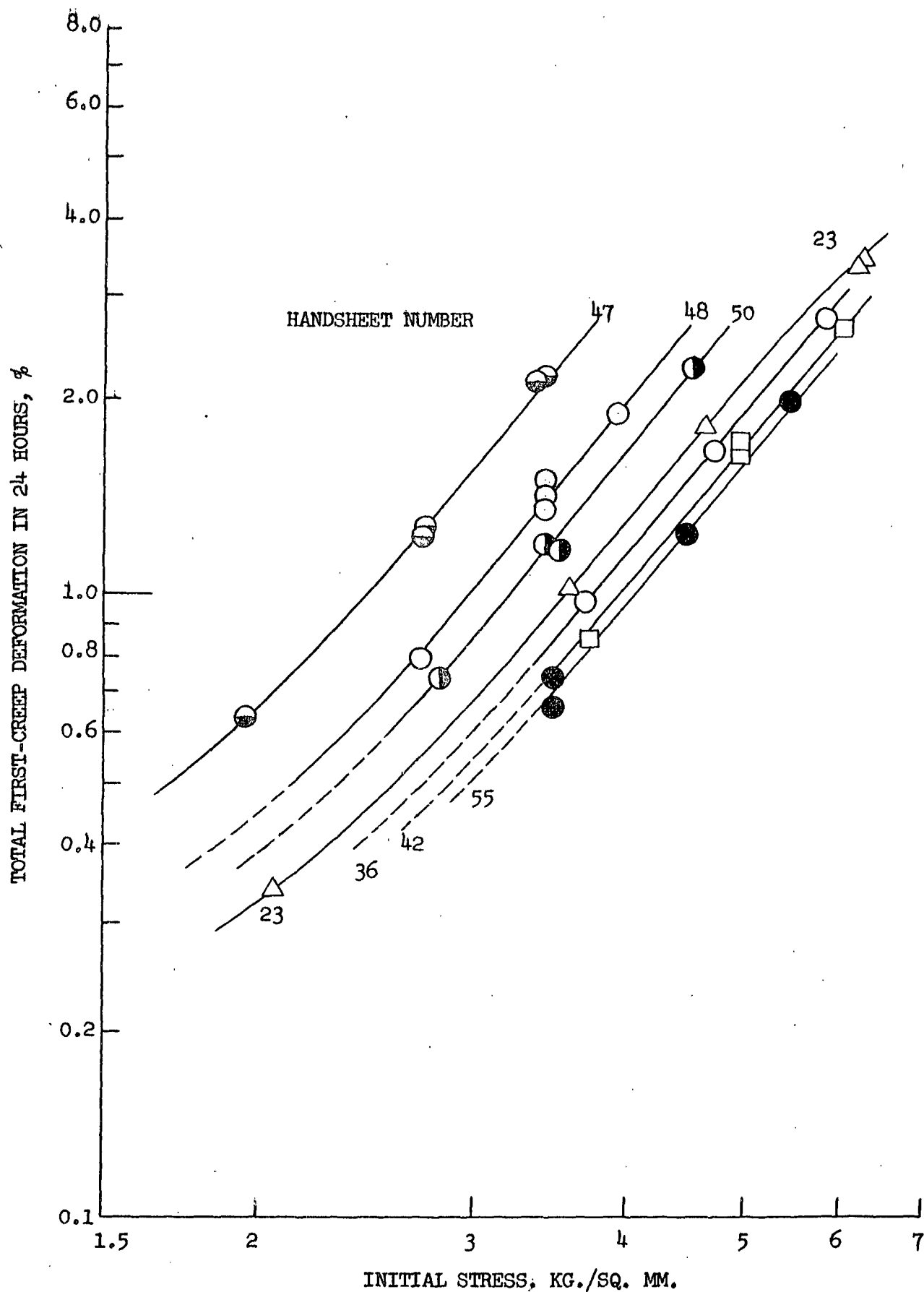
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TABLE XIV (Continued)

SUMMARY OF FIRST-CREEP AND FIRST-RECOVERY TESTS
ON HANDSHEETS OF DIFFERENT SOLID FRACTION

Relative Humidity, 50%
Temperature, 73°F.

Test No.	Spec. No.	Handsheet Solid Fraction, %	Load, kg.	Initial Stress, kg./sq. mm.	Total 24-Hr. First-Creep Deformation, %	Total 24-Hr. First-Recovery Deformation, %
34	28-4	45.6	3.50	3.62	1.05	- -
35	29-3	45.1	3.50	3.74	1.08	- -
36	30-11	44.9	3.50	3.65	1.01	- -
37	31-9	44.5	3.50	3.61	0.99	0.62
38	32-8	45.8	3.50	3.69	0.99	- -
39	33-8	46.7	3.50	3.64	1.03	0.63
86	36-5	51.7	3.50	3.70	0.97	0.63
87	36-3		4.50	4.71	1.68	0.83
91	36-9		4.50	4.71	1.69	0.84
88	36-7		5.50	5.82	2.79	1.04
118	42-9	54.2	3.50	3.80	(0.85)	- -
129	42-1		4.50	4.90	1.68	- -
104	42-4		4.50	4.90	1.72	- -
115	42-6		5.50	5.95	(2.62)	- -
78	55-7	51.7	3.50	3.49	0.65	0.48
90	55-4		3.50	3.48	0.73	0.53
77	55-10		4.50	4.48	1.23	0.72
79	55-12		5.50	5.45	2.03	0.93



curves are approximately linear between total first-creep deformations of about 0.6 to 2.5% and nearly parallel for all handsheets. Parallelism between the curves of different handsheets would also exist in log-log plots of the total first-creep strain per unit initial stress versus initial stress. Thus, changes in solid sheet structure by beating and wet pressing are noted in Figure 20 as shifts of the curves along the log-stress axis. If widely different test durations are considered, however, slight deviations from parallelism may occur since the first-creep curves were not identical in shape for all handsheets. The deformation-time data for these tests are given in Table A of the Appendix. Plots of these data (not shown) indicated rough agreement in shape of the first-creep curves of all handsheets when compared at equivalent levels of total first-creep deformation. Generally, it was noted that the first-creep curves of handsheets prepared from lower freeness pulps tend to be flatter at early times and to rise more sharply at later times. Since the differences in curve shape were small, these data were inadequate to explore these effects in detail. The chief differences in the first-creep behavior of these handsheets lies in the widely different initial stresses required to reach specified total first-creep deformations in equal-duration tests.

Master creep curves could be constructed from the first-creep curves of all handsheets by the technique used for Handsheet 23. The load effect, therefore, is basically the same at all solid fractions and degrees of beating. As a first approximation, the master creep curves of all handsheets were similar in shape, but were shifted relative to each other along both the deformation per unit initial stress and the log-time axes. The timeshift requirements for the construction of the master creep curves

are summarized in Table XV along with the estimated apparent elastic moduli and the K/S_0 values for the logarithmic creep portions of the first-creep curves. The time shift versus stress relationships were linear for all handsheets. A time shift of approximately 2.2 decades of log time per unit initial stress was required for all handsheets prepared from 775 cc. S.-R. freeness pulp compared to about 1.45 decades of log time per unit initial stress for all other handsheets. The time-shift requirement was not a function of solid fraction, but appears to be related in some manner to the degree of beating. No difference in time-shift requirement, however, was noted between handsheets prepared from 620 and 425 cc. S.-R. freeness pulp. The only experimental evidence for the time shift is the empirical fitting of the reduced first-creep curves. Based on the continuity in shape of the reduced curves when shifted along the time axis, one may postulate that the time shift is necessary because of a decrease in the various retardation times with increasing initial stress. Since the time-shift requirement was not a function of solid fraction, one must further assume that the structural factors which affect the postulated changes in retardation time are related chiefly to the molecular structure of the polymer. After mechanical conditioning, the time-shift requirement is much smaller compared to the requirement in first-creep tests. If this effect could be related to a greater degree of molecular order after mechanical conditioning, then the smaller time-shift requirements for lower freeness pulps might also be attributed to a more orderly molecular structure. Since the time-shift requirements changed with beating but not with wet pressing, it might further be postulated that enhanced swelling of the fibers with greater beating provided for greater changes

TABLE XV
FIRST-CREEP PROPERTIES OF HANDSHEETS
OF DIFFERENT SOLID FRACTION

Relative Humidity, 50%
Temperature, 73°F.

Handsheet Number	Estimated E_a , kg./sq. mm.	Solid Fraction, %	Initial Stress, kg./sq. mm.	Shift in Log Time to Form Master Creep Curve	K/S_0 sq. mm./kg. x 10,000
47	700	31.6	1.95	3.15	10.36
			2.72	1.70	
			2.73	1.60	
			3.43	0.0	
			4.39	2.35	
48	800	40.6	2.73	1.65	8.52
			3.43	0.25	
			3.43	0.0	
			3.92	0.95	
50	860	31.6	2.81	1.0	8.17
			3.53	0.0	
			4.53	2.65	
52	1100	39.9	2.69	1.80	6.79
			3.30	0.0	
			4.27	1.70	
23	1000	47.3	2.06	2.20	7.02
			3.60	0.0	
			4.62	1.70	
			5.70	2.80	
			6.21	3.75	
36	1050	51.7	3.70	0.0	7.02
			4.71	1.45	
			4.71	1.55	
			5.82	3.20	
42	1100	54.2	3.80	0.0	6.91
			4.90	1.95	
			5.95	3.35	
55	1150	51.7	3.49	0.0	7.37
			4.48	1.55	
			5.45	2.90	

in molecular orientation during drying of the handsheets. The time shift required to obtain coincidence of any two reduced first-creep curves, however, is extremely sensitive to curve shape. It is possible that the different time-shift requirements are related in part to the small differences in curve shape that exist. Much more work would be required to investigate this possibility.

The relationship between solid fraction and total first-creep deformation in 24 hours at an initial stress of 3.75 kg./sq. mm. is shown in Figure 21. These data were obtained from the curves of Figure 20 and are summarized in Table XVI. In handsheets prepared from 775 cc. S.-R. freeness pulp (47, 48, and 50), the creep response is large at solid fractions in the order of 30%, but decreases rapidly with increasing solid fraction and becomes less dependent on solid fraction at higher values. At lower pulp freenesses, the deformations are lower and should describe similar curves displaced largely along the deformation axis. In any event, beating causes a reduction in total first-creep deformation at comparable solid fractions.

TABLE XVI

TOTAL FIRST-CREEP DEFORMATIONS AT 3.75 KG./SQ. MM.
FOR HANDSHEETS OF DIFFERENT SHEET STRUCTURE

Handsheet Number	Solid Fraction, %	Total First-Creep Deformation in 24-Hours At 3.75 kg./sq. mm., %
47	31.6	2.70
48	40.6	1.70
50	50.6	1.45
52	39.9	1.38
23	47.3	1.12
28-33	45.4	1.1
36	51.7	1.00
42	54.2	0.84
55	51.7	0.80

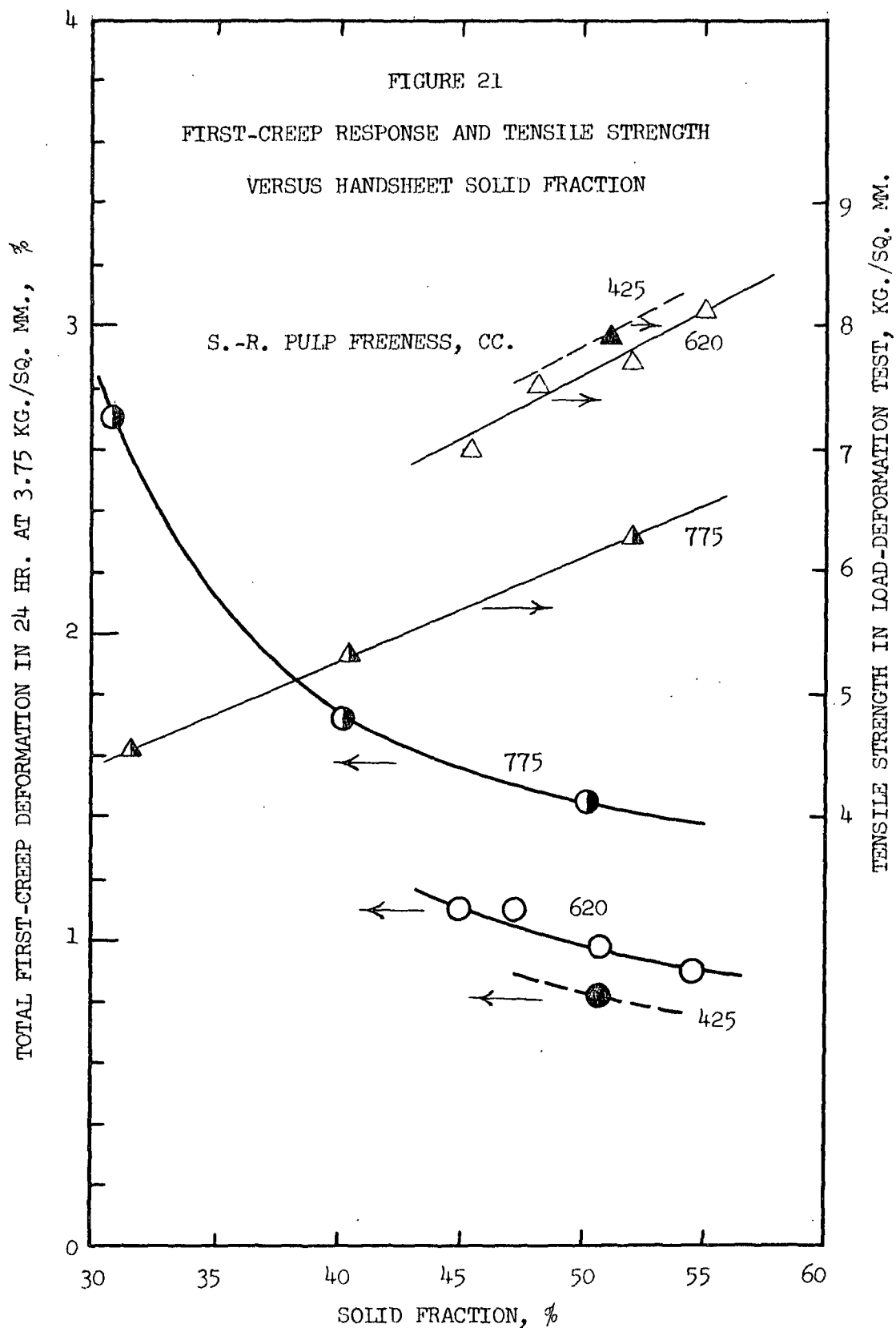


Fig. 21. First-Creep Response and Tensile Strength
Versus Handsheet Solid Fraction

The specific scattering coefficients of Table II suggest that a given increase in solid fraction in the lower range involves small increases in percentage of optical bonded area, whereas rather large increases in percentage of optical bonded area occur in later increments of increasing solid fraction. Thus, the early increases in percentage of optical bonded area appear to be much more effective in reducing the total creep deformation than are later increases. Tensile strength data of load-deformation tests are also shown in Figure 21 to illustrate that strength increases almost linearly with solid fraction in the range where the creep response versus solid fraction curve is nonlinear. These tensile strength data are averages of relatively few tests for all handsheets within the groups of Table II. Forman (59) indicated that linear relationships between tensile strength and solid fraction, e.g., apparent sheet density, are common. It is postulated that an early increase in solid fraction with relatively small increases in bonded area include many new fiber-fiber contacts. The deformation at specified initial stresses in given periods of time will be highly dependent on the stress distribution both between and within the fibrous elements. An increase in the number of interfiber contacts would have a large effect on the stress distribution. Later increases in solid fraction are attributed to continued increases in interfiber bonded area with fewer new interfiber contacts, which may have less influence on the stress distribution pattern throughout the sheet. Sheet strength continues to increase but the response to stress is less sensitive to these later increases in bonded area and solid fraction.

The creep response of Handsheet 52, prepared from the long-fibered fraction of 775 cc. S.-R. freeness pulp, is lower than the response of any

of the handsheets prepared from the unscreened pulp. The solid fraction and tensile strength were comparable to those of Handsheet 48 which was wet pressed at the same conditions. This agrees with the observations of Haywood (60) for screened oak pulp. The reduced creep response poses a problem in interpretation. The apparent elastic modulus was high and the slope of the logarithmic-creep portions of the first-creep curves was lower than that of any other handsheet of this series at equivalent initial stresses. The time shift to form the master creep curve was comparable to that of any 775 cc. S-R. freeness pulp. One fact seems certain. The interfiber bonded area should not exceed that of Handsheet 48, although it cannot be assumed that the bonded area will be significantly lower. By removal of the fines, a greater number of long fibers should be found in a given solid cross sectional area of the specimen. It seems probable that a sheet containing a greater percentage of long fibers could have a different pattern of stress distribution in tensile loading, and that this could account for the observed creep behavior of Handsheet 52. With these few data, however, one can only speculate regarding the reasons for the reduced creep response in this handsheet. Further work is needed to establish the sheet structure to creep property relationships of handsheets prepared from various screened pulp fractions.

FIRST-RECOVERY PROPERTIES

Time-deformation data in first-recovery tests are given in Table A of the Appendix. The first-recovery curves are not shown here. A comparison, however, of the first-recovery curves of all tests at all initial stresses indicated that these curves were identical in shape when compared at equivalent levels of total first-recovery deformation. If this similarity in curve shape were applicable over the entire time interval,

starting with the instant of load removal, a relationship between the immediate elastic deformation and the delayed elastic deformation in recovery tests may exist which is independent of initial stress. As a first approximation, the curves relating the total first-recovery deformation to the initial stress for the various handsheets could be described by a family of parallel straight lines on a log-log plot. The same order that existed in total first-creep deformation versus initial stress plots prevails.

Possibly the most significant effect in the first-recovery behavior of the various handsheets lies in the relationship between the total first-recovery and the total first-creep deformations in 24-hour tests as shown in Figure 22. It is of particular significance that the recovery deformations of all handsheets fell within such a narrow range at given total first-creep deformations. Handsheets 47, 48, and 50 have almost identical recovery versus creep relationships despite the fact that the solid fraction ranges from 31.6 to 50.6%. The recovery was slightly greater for Handsheets 36 and 55 and slightly less for Handsheet 52. The importance of these relationships cannot be overemphasized. These curves offer strong evidence that the mechanisms of response are of the same type at all solid fractions and that the nonrecoverable deformation is related to the total first-creep deformation at any specified test duration. The principal differences in creep behavior lie in the different stresses required to reach specified deformations. This evidence discounts any hypothesis which proposes that the nonrecoverable deformation is due to mechanisms of response which are unrelated to the recoverable deformation. Rather, these data are consistent with the concept that the rate-

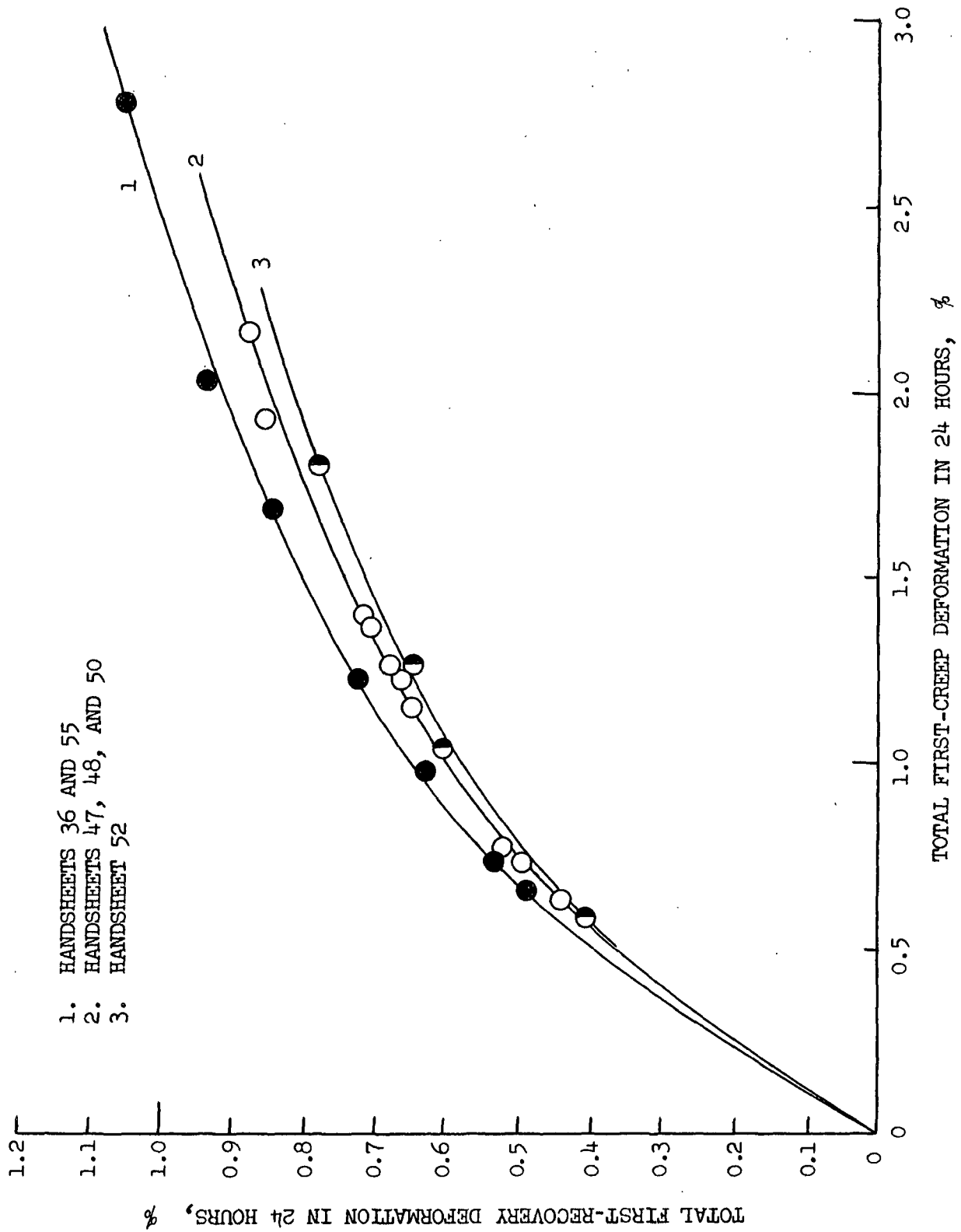


Fig. 22. Total First-Recovery Deformation Versus Total First-Creep Deformation in 24-Hour Tests

controlling mechanisms of response in both first-creep and primary-creep response are due directly to molecular structural changes. It is highly improbable that one could attribute the first-creep response of paper to several different mechanisms of response which vary with solid fraction in different ways and still obtain data of the type shown in Figure 22.

The differences in percentage recovery at any first-creep deformation may be related to beating but not to solid fraction. At equal total first-recovery deformations in 24 hours, the total first-creep deformation is smallest for the more highly beaten pulp. It would seem that with greater beating, the handsheet is partially mechanically conditioned compared to handsheets prepared from less beaten pulps. This is the same effect which is expected with increased tension during drying. Since solid fraction does not materially affect the percentage of recovery, it may again be postulated that greater drying tensions under conditions of nonshrinkage in the plane of the sheet occur with greater swelling of the fibers in beating. It is expected that changes in drying tension or extent of shrinkage during drying will markedly affect the relationship between first-recovery and first-creep deformation.

In summation, the results of this series of tests demonstrate that the recovery curves obtained on handsheets of different solid fraction are of similar shape when compared at equivalent levels of total first-recovery deformation, but that the stress required to reach specified total first-recovery deformations varies widely with solid fraction. The principal effect of solid fraction on both creep and recovery response may be described by shifts of the log-deformation versus log-initial stress

curves along the log-initial stress axis. The first-creep curves obtained on handsheets of different solid fraction differed slightly in shape when compared at equivalent levels of deformation. In all cases, however, the first-creep curves of any handsheet could be combined to form a master creep curve by stress-proportional reductions in deformation plus stress-proportional shifts of the reduced curves along the log-time axis. Greater time shifts per unit initial stress, greater nonrecoverable deformations at equal total first-creep deformations, and greater first-creep response at constant initial stress and solid fraction occurred with lesser degrees of beating.

CREEP PROPERTIES VERSUS RELATIVE HUMIDITY

Water is a natural plasticizer of paper and other cellulosic materials. Any interpretation which might be applied to the relation between paper properties and structure must consider both the effect of the existing moisture content and the previous moisture history. Stamm (61) reviewed the many hypotheses concerning the mechanisms of bonding between cellulose and water. In the general case, the action of a plasticizer in affecting mechanical properties is due to the disruption and weakening of the bonding forces between polymer molecules and to the physical separation of those molecules preventing polymer-polymer contacts (62). A reduction in the number of intermolecular bonds will tend to decrease the order of the molecular configuration and may increase the amount of response. In addition, easier extensibility may occur with increasing plasticizer content because of a decrease in retardation times.

One seeks ultimately to define the effect of a plasticizer in terms of its effect on the individual mechanisms of deformation. At the same time, however, it becomes possible to use the relation between plasticizer content and mechanical behavior as a means of separating and possibly defining the various mechanisms of response. This is the specific aim of this investigation of the influence of relative humidity.

EXPERIMENTAL PROCEDURES

Handsheets 28 through 33 were selected for this series of tests. A comparison of first-creep deformations at a single value of load on a single specimen of each handsheet indicated a satisfactory level of precision between handsheets. This was important since the number of replicate tests had to be held to a minimum in this work (see Table XIV).

The testing procedures were as follows. In all cases, the specimens were inserted in the creep testing units at 50% R.H. to an initial length of 10.00 inches in the as-dried condition. That condition is drying-desorption to 50% R.H. and storage at $50\% \pm 3\%$ R.H. The initial specimen width was 1.00 inches. An initial micrometer reading was obtained at 50% R.H. and the relative humidity was changed to the desired values. In tests at 23.5, 63, 73.5, and 83% R.H., the initial change in relative humidity was to 12% for 48 hours followed by a second change to the test humidity. A minimum period of 48 hours or an estimated maximum change in length of less than 0.02% in 24 hours was allowed before the specimens were tested. The specimens at 50% R.H. were run in the as-dried condition and those tested at 94% R.H. were brought to that

relative humidity by adsorption directly from 50% R.H. All of the specimens were aged for several months prior to testing and it was established that the results of testing in the as-dried condition at 50% R.H. would not be significantly different from tests at 50% R.H. reached by adsorption from 12% R.H. This may have been due in part to the long period of aging at 50% R.H., which lowered the as-dried moisture content to about 7.4% compared to a desorption moisture content of about 8.0% and an adsorption moisture content of about 6.8% on the oven-dry basis. It was felt that mechanical properties at 94% R.H. would be insensitive to whether that humidity was reached by adsorption from 50 or 12% R.H.

The specimen length changed slightly with relative humidity. The widths and solid cross-sectional areas would also change with relative humidity. These data were treated, however, on the basis of a constant amount of solid fibrous material between the clamps. Initial stresses were calculated as the ratios of creep loads to the calculated solid cross-sectional areas of the specimens at 50% R.H. The deformations are measured in all cases from the specimen length at the start of each test and considered always as a percentage of the 10.00-inch initial specimen length at 50% R.H. The actual initial specimen lengths were not given in these data, but may be estimated from the hygroexpansivity data of Table XXV.

A specimen of Handsheet 21 was suspended in the conditioned atmosphere from an analytical balance during each test. The weights of these specimens were recorded during the course of the test and the specimens were oven dried at the end of the test to determine the moisture content. Reported moisture contents are considered accurate to $\pm 0.3\%$.

The relative humidity and temperature were recorded at intervals throughout the course of the tests. Relative humidity was measured with

electric hygrometer elements calibrated over saturated salt solutions at 73°F. using the equilibrium relative humidity values reported by Wink (53). Fluctuations in relative humidity fell within a total range of 2% during the course of the tests; however, the values are reported to the nearest 0.5% and represent the most frequent measurement. The temperature varied between 72.5 and 73.5°F. throughout the course of these experiments, but was less variable during any given test.

The conditions of relative humidity and moisture content pertinent to this series of tests are summarized in Table XVII.

TABLE XVII

SUMMARY OF TEST CONDITIONS
Temperature, 72.5 to 73.5°F.

Remarks	Saturated Salt Solution for Humidity Control	Equilibrium R.H. at 73°F., %	Measured R.H., %	Moisture Content, % (ovendry basis)
Preconditioning	Lithium chloride	11.1	12	3.0
Tests 71-75	Potassium acetate	22.9	23.5	4.6
Tests 37,39,133-136	--	--	50 ^a	7.4 ^b
Tests 51-56	Sodium nitrite	64.8	63	8.5
Tests 80-85	Sodium chlorite	75.5	73.5	10.4
Tests 40-45	Potassium chromate	86.5	83	14.0
Tests 127-129,132	Potassium sulfate	97.8 (63)	94	21

^a Room conditions

^b Approximate moisture content of as-dried specimens at 50% R.H.

RESULTS AND DISCUSSION

A summary of the tests in this series is presented in Table XIX. The discussion and presentation of results which follow are based solely on these tests. Further discussion is presented in the following section regarding the effect of humidification on creep behavior. It is necessary to consider both sections to gain a more complete account of the effect of relative humidity on the creep properties of alpha-pulp handsheets.

APPARENT MODULUS OF ELASTICITY

Estimates of the apparent modulus of elasticity from measurements of the reversible deformations during the rapid application and removal of small loads were generally inaccurate at relative humidities above 50% R.H. It was possible in some cases to calculate the immediate elastic deformation in first-creep tests at lower initial stresses where the early response could be fitted to an exponential equation. The estimated values presented in Table XVIII are taken largely from creep curves at low initial stresses.

TABLE XVIII

ESTIMATED APPARENT ELASTIC MODULI VERSUS RELATIVE HUMIDITY

Relative Humidity, %	Elastic Modulus, kg./sq. mm.
23.5	1090
50	1050
63	970
73.5	840
83	700
94	450

The apparent modulus of elasticity is a weak function of relative humidity below about 60%, but decreases rapidly at higher relative humidities. These data suggest that the apparent modulus of elasticity will approach extremely low values at 100% R.H.

FIRST-CREEP RESPONSE

Master Creep Curves

It had been shown earlier for Handsheet 23 in tests at 50% R.H. that the various first-creep curves could be combined into a single

TABLE XIX

SUMMARY OF TESTS AT VARIOUS RELATIVE HUMIDITIES
Adsorption Series

Temperature, 72.5 to 73.5°F.

R.H., %	Test No.	Spec. No.	Load, kg.	Initial Stress, kg./sq. mm.	Total 24-hr. First-creep Deformation, %	R.D.D., ^a %	Total 24-hr. First-Recovery Deformation, %
23.5	74	31-6	2.80	2.93	0.46	0.17	0.37
23.5	73	30-4	3.50	3.68	0.65	0.27	0.48
23.5	72	29-4	3.50	3.76	0.69	0.30	0.51
23.5	76	32-7	4.50	4.71	1.04	0.51	0.67
23.5	75	33-3	4.50	4.67	1.06	0.52	0.67
23.5	71	28-7	5.50	5.69	1.86	1.01	0.93
50	134	30-1	2.80	2.96	0.59	0.26	0.46
50	136	33-1	3.50	3.62	0.93	0.49	0.61
50	133	33-9	4.50	4.65	1.94	1.12	0.89
50	135	32-4	5.00	5.21	2.25	1.29	0.95
63	51	28-1	2.00	2.09	0.59	0.32	0.39
63	53	31-8	2.80	2.91	1.10	0.67	0.60
63	54	32-9	2.80	2.95	1.13	0.69	0.62
63	52	30-3	3.50	3.68	1.77	1.09	0.78
63	56	32-11	3.50	3.68	1.83	1.14	0.80
63	55	33-4	4.50	4.67	3.10	1.73	1.05
73.5	84	32-5	2.00	2.10	0.82	0.52	0.48
73.5	83	31-1	2.80	2.94	1.52	0.98	0.72
73.5	82	30-7	2.80	2.91	1.62	1.03	0.73
73.5	85	33-2	3.50	3.61	2.39	1.40	0.90
73.5	81	29-2	3.50	3.73	2.55	1.45	0.92
73.5	80	28-6	4.00	4.12	3.19	1.66	1.03
83	43	32-2	2.00	2.09	1.28	0.83	0.63
83	44	28-2	2.00	2.07	1.43	0.95	0.67
83	42	31-7	2.80	2.91	2.49	1.45	0.93
83	40	32-3	3.50	3.65	3.02	1.54	1.04
83	41	30-5	3.50	3.68	3.24	1.55	1.07
83	45	33-6	3.50	3.62	3.77	1.66	1.15
94	132	28-9	0.80	0.82	0.80	0.55	0.41
94	129	28-10	1.30	1.34	1.59	1.03	0.73
94	127	30-6	2.00	2.09	2.66	1.34	1.04
94	128	30-8	2.50	2.60	3.66	1.43	1.25

^a Relative delayed deformation between 10 and 86,400 seconds in first-creep tests.

generalized or master creep curve by stress-proportional reductions in deformation plus stress-proportional shifts along the log time axis to obtain coincidence in the regions of overlap. This technique effectively described the relationship between initial stress and first-creep response. The creep behavior of the handsheets used in this series of tests agree well at 50% R.H. with the results determined for Handsheet 23; hence, only the curves at the other relative humidities will be considered at this time.

The first-creep curves plotted as total first-creep strain per unit initial stress versus log time are shown in Figures 23 through 27. It was possible to form master creep curves with excellent agreement in the regions of overlap by shifting the curves at 23.5 and 63% R.H. along the log time axis. At 73.5% R.H., disagreement was noted in the regions of overlap. The disagreement became greater at higher relative humidities. Its nature is readily noted in the reduced creep curves at 94% R.H. The deformation versus log time plot at 94% R.H. is shown in Figure 28. The break in these curves was due to a steady decline in the relative humidity which was corrected at the point of the sharper rise in response. Sufficient time elapsed after this fluctuation in relative humidity to permit the response to approach the proper deformation-time relationship during the latter part of these tests. Better agreement in the regions of overlap could be obtained by using deformation reduction factors which are nonlinearly related to initial stress. At 94% R.H., reasonable agreement would be obtained by merely shifting the creep curves of Figure 28 without any prior reduction in deformation. The selection of appropriate deformation reduction factors may not enable the accurate construction of master creep curves since it appears that the shape of the first-creep curve may become sigmoidal at intermediate

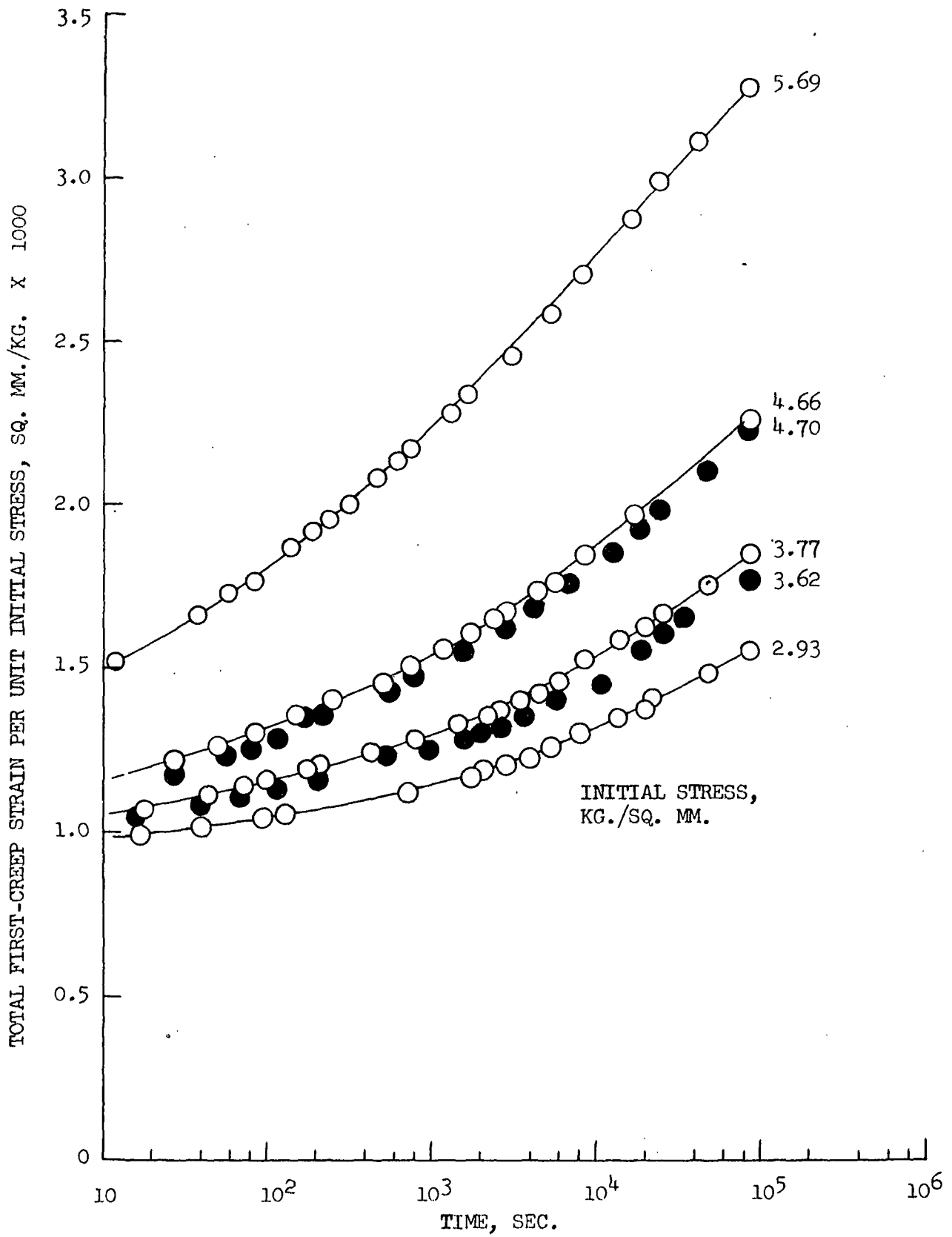


Fig. 23. First-Creep Curves at 23.5% R. H.

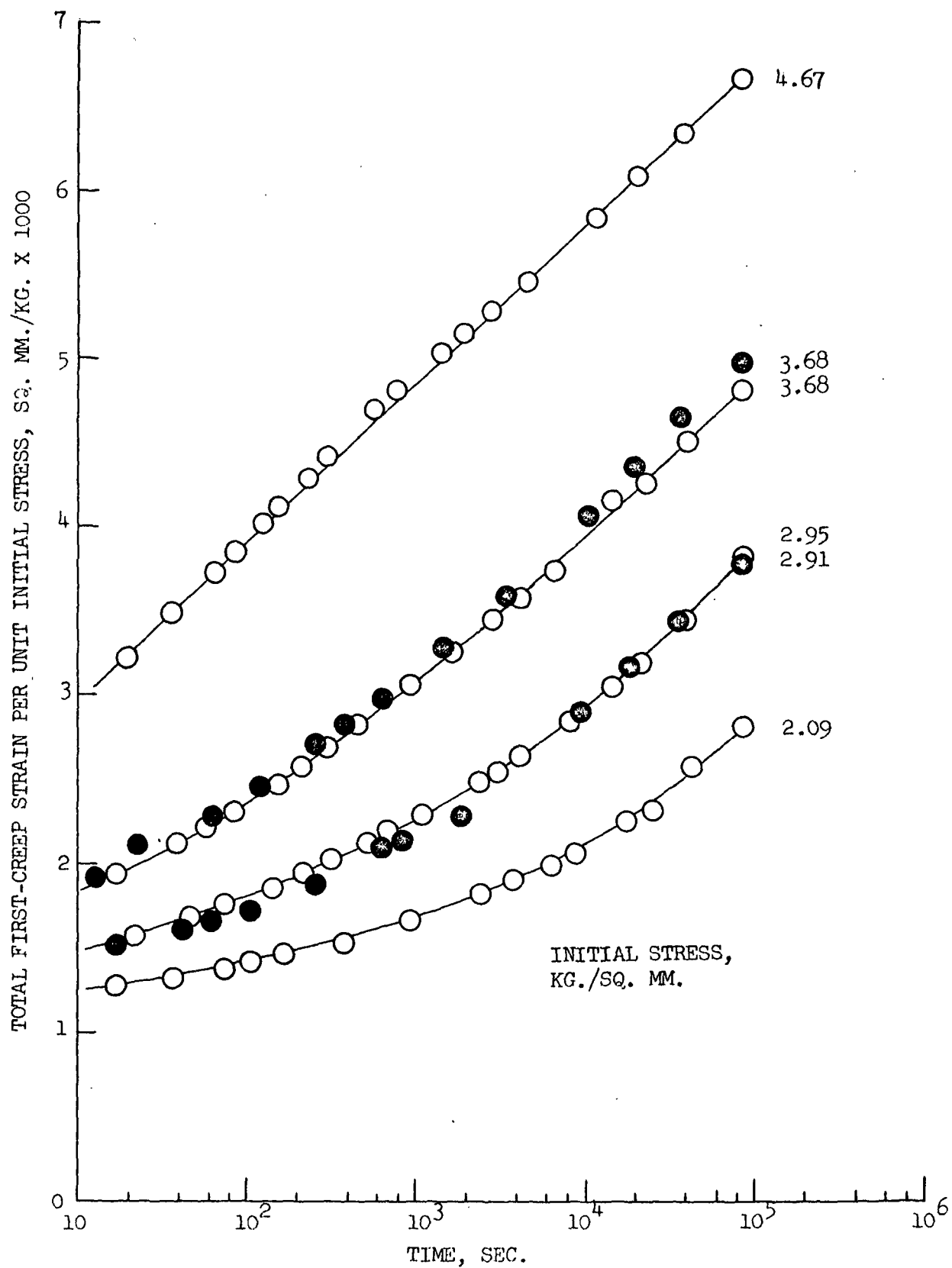


Fig. 24. First-Creep Curves at 63% R.H.

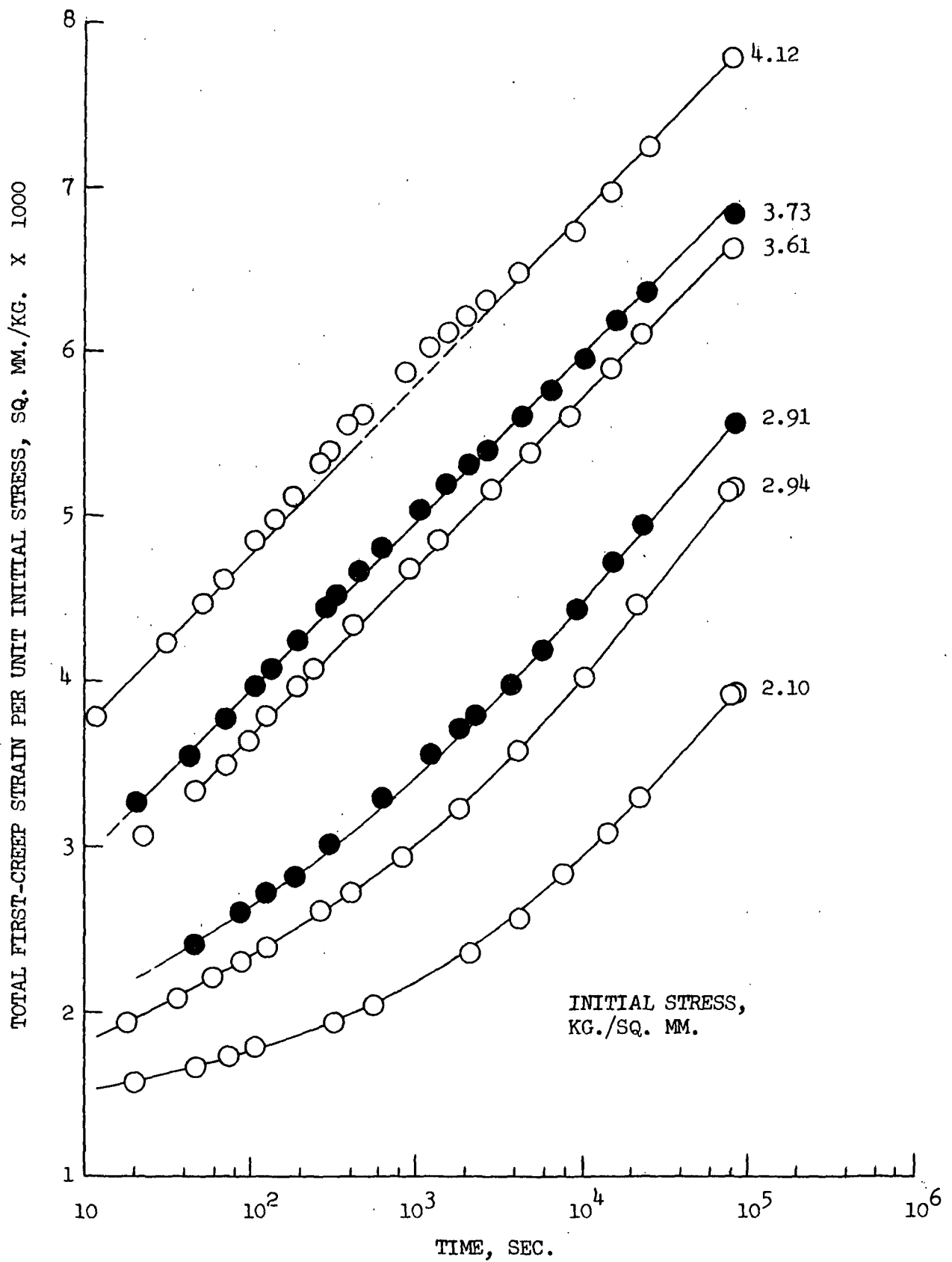


Fig. 25. First-Creep Curves at 73.5% R.H.

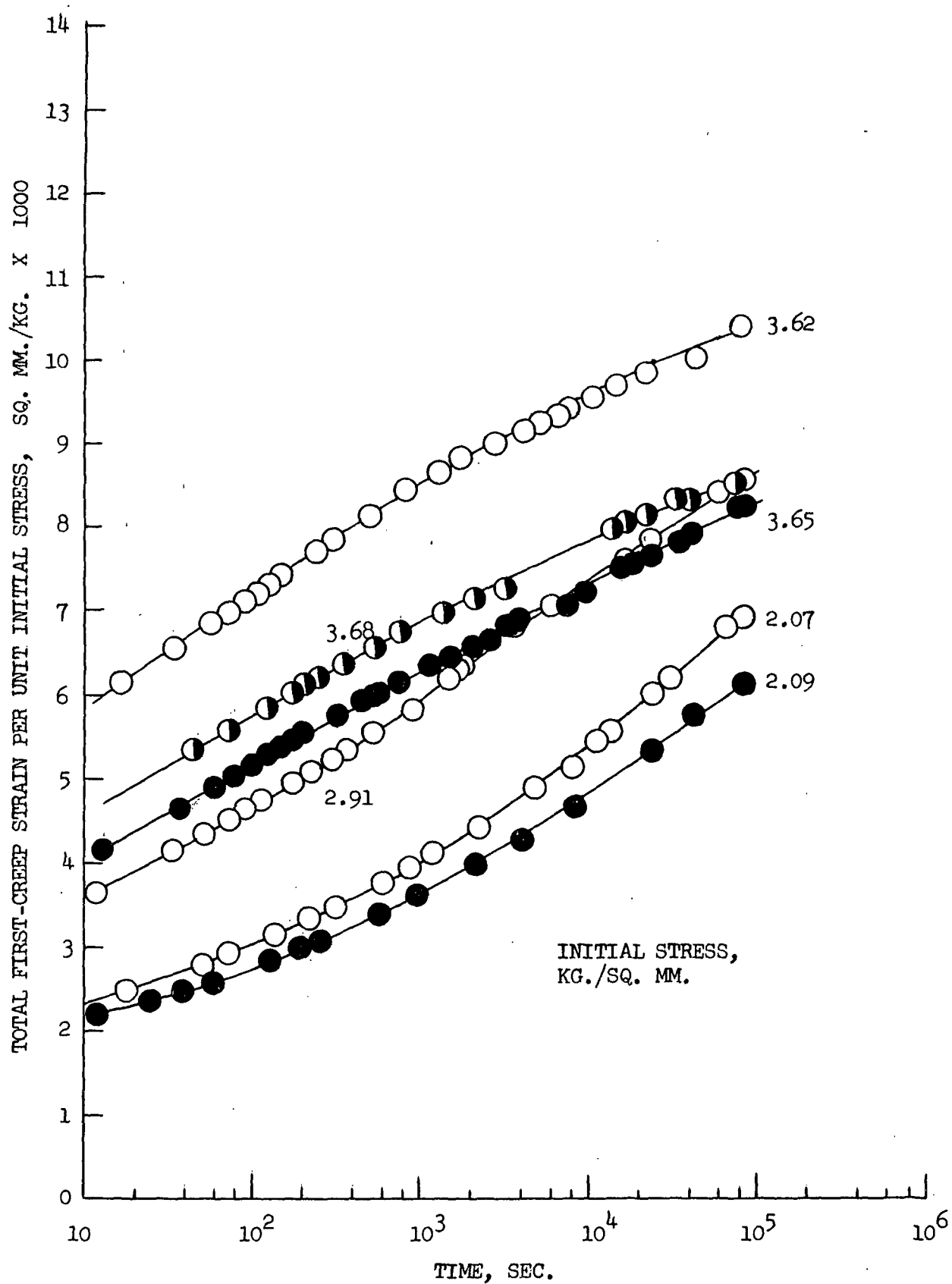


Fig. 26. First-Creep Curves at 83% R.H.

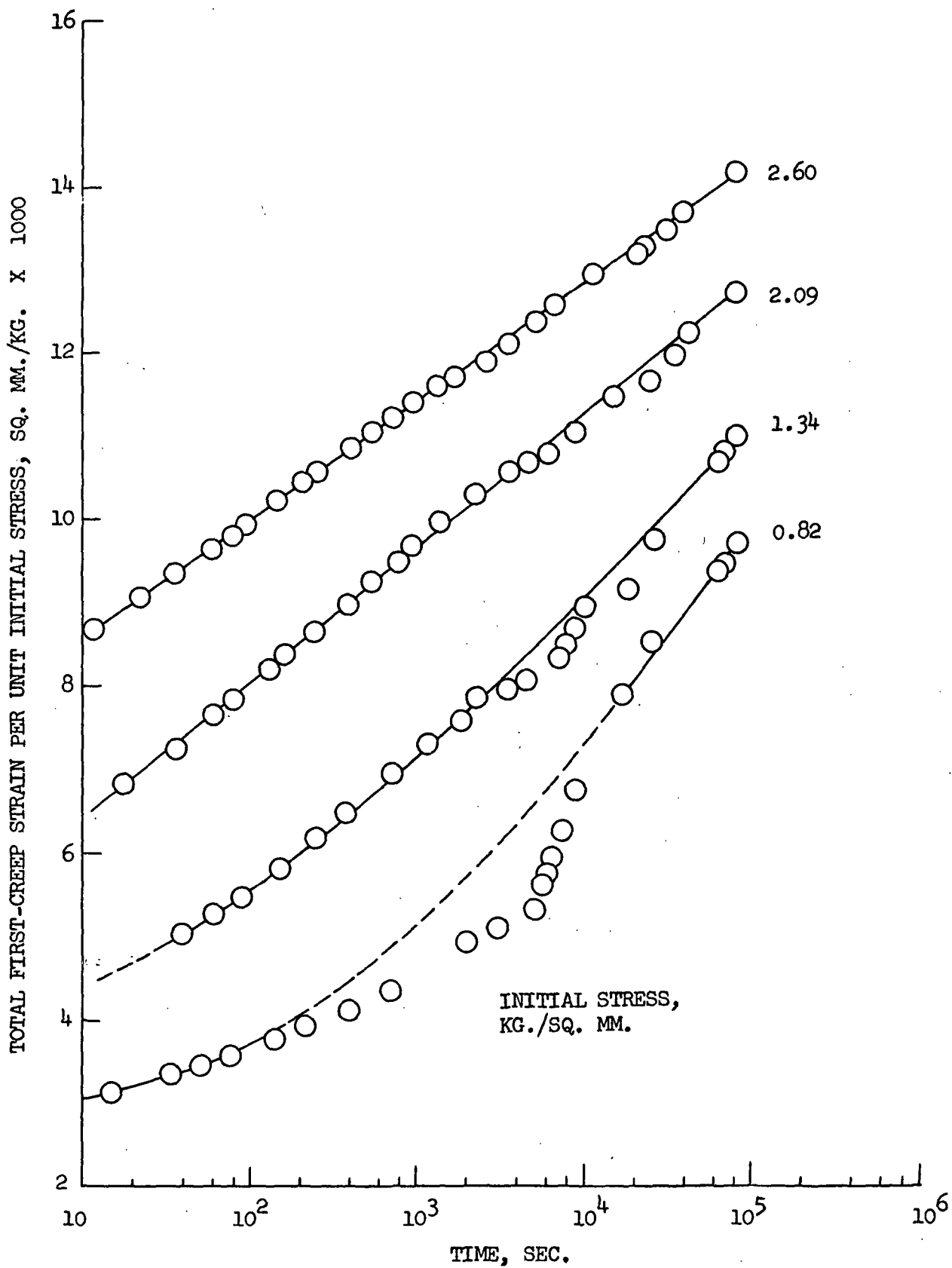


Fig. 27. First-Creep Curves at 94% R.H.

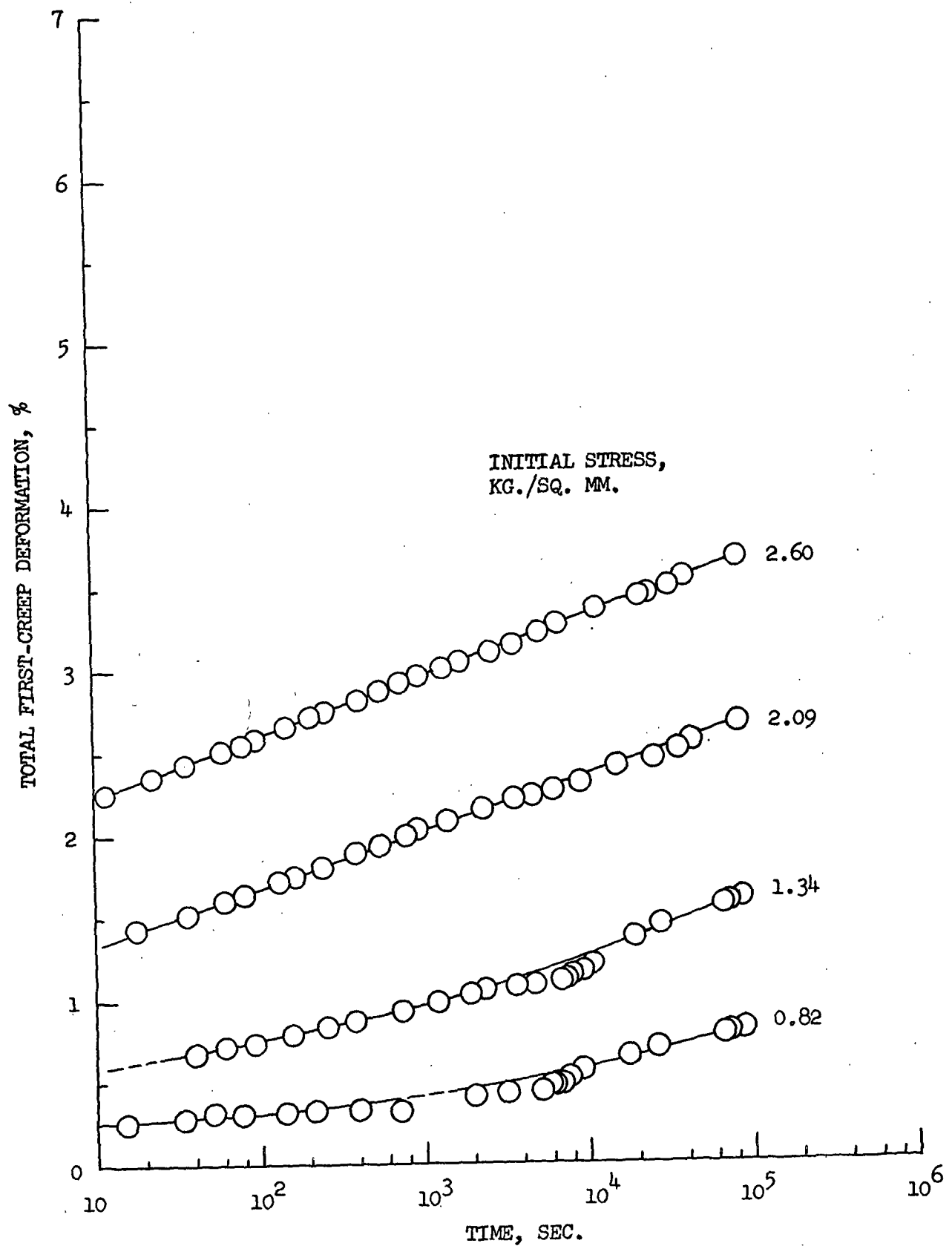


Fig. 28. First-Creep Curves at 94% R.H.

and possibly at higher initial stresses. Note particularly the curve at 83% R.H. and 2.91 kg./sq. mm. This type of behavior was more evident in tests following humidification of these specimens where logarithmic creep was approached by a concave-downward curve at higher relative humidities and initial stresses in contrast to the concave-upward curves common to tests at lower relative humidities.

The relationship between the required timeshift and initial stress could be determined at 23.5, 50, and 63% R.H. At 23.5% R.H., the relation between time shift and initial stress was linear within an allowable experimental error of about 8% in total first-creep deformation. The trend, however, was toward a curved relationship with small time shifts at low stresses, which increase with increasing initial stress. Linearity was noted at 50 and 63% R.H. Since small errors or variations in total first-creep deformation can cause large differences in the required time shift, these data must be regarded as estimates. The estimated time-shift requirements expressed as decades of log time per unit initial stress were about 1.2 at 23.5% R.H., 1.5 at 50% R.H., and 1.6 at 63% R.H. At 73.5% R.H., the requirement increased to 1.8 and it appears that with increasing relative humidity, the time-shift requirement will increase further. For example, if the creep curves at 94% R.H. were reduced properly in deformation to form a master creep curve, the time shift requirement would be over 4 decades of log time per unit initial stress.

It was assumed in a previous discussion that the time-shift requirement may be proportional to the degree of molecular disorder of the polymer. These data do not contradict that assumption, but are insufficient to permit an adequate interpretation of the relationship between the required time shift and structure.

Deformation Time-Initial Stress Relationships

The total 24-hour first-creep deformations are plotted versus initial stress at various relative humidities in Figure 29. This family of curves bears a close resemblance to curves relating the same variables at constant relative humidity with time as a parameter (see Figure 7). At least one of the effects of increasing relative humidity is a speeding up of the response at constant initial stress. This is a good first approximation for the earlier exponential response, since little difference existed in the value of the exponent a of exponential equations between 23.5 and 73.5% R.H. In this range of relative humidity, the exponent remained relatively constant at 0.23. The creep response at the higher relative humidities was not in the proper range to permit calculation of the constants of exponential equations. Changes in the other constants of exponential equations have the effect of shifting the curves along the deformation and time axes. This type of behavior is suggestive of reductions in the energy of activation for delayed configurational elastic response with increasing moisture content. It is somewhat analogous to a shift in creep response along the log-time axis with increasing temperature, which is common for many plastics. There are, however, some other prominent effects at higher relative humidities, which relate chiefly to the manner in which creep response is affected by increasing initial stress. These were indicated by the inability to form a master creep curve at the higher relative humidities, and involve a tendency toward stress independence in the long-duration response at higher initial stresses. This is illustrated by plots of the relative delayed deformation between 10 and 86,400 seconds versus initial stress at the various relative humidities (Figure 30). At 83 and

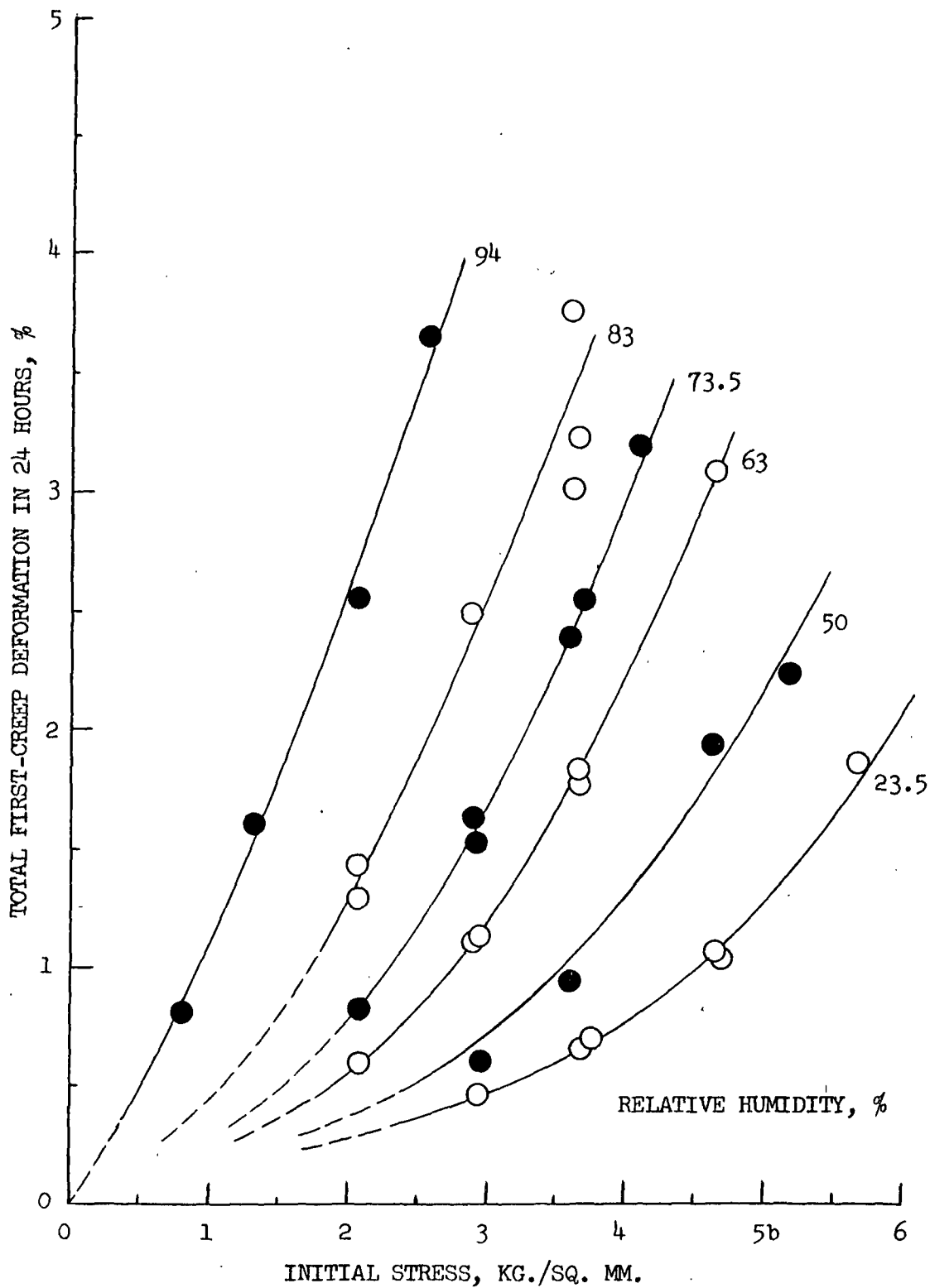


Fig. 29. Total First-Creep Deformation Versus Initial Stress
at Various Relative Humidities
in 24-Hour Tests

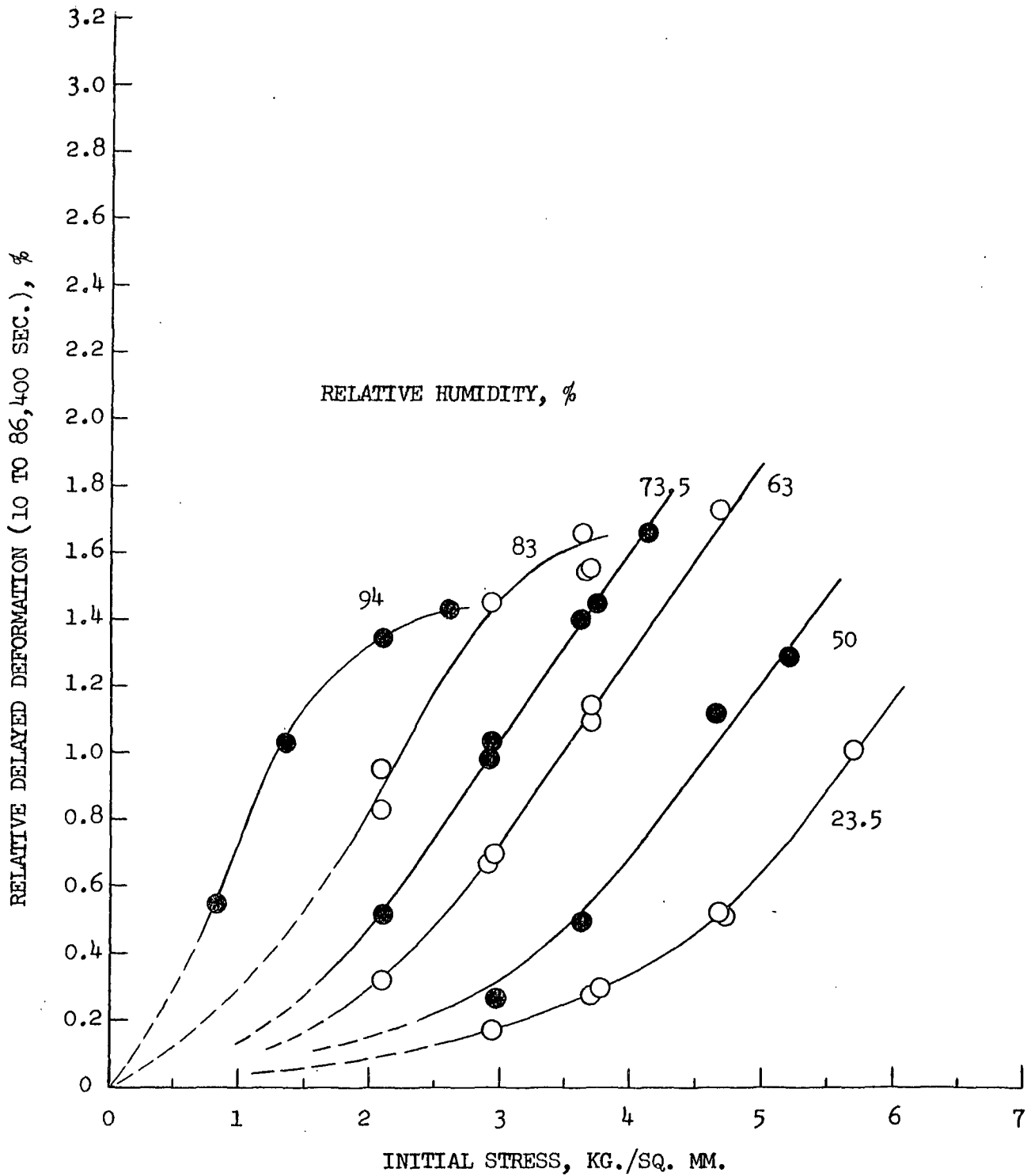


Fig. 30. Relative Delayed Deformation Between 10 and 86,400 Seconds
Versus Initial Stress at Various Relative Humidities
in First-Creep Tests

94% R.H., the continued increase in total first-creep deformation occurred largely before 10 seconds at the higher initial stresses. It has been shown earlier that the same effect occurs in the delayed primary creep response at 83% R.H.

A creep test can be described in part by stating the stress, time, or deformation with the other two variables held constant. Such single-valued descriptions are useful in illustrating the effect of variables in structure or external test conditions on creep response. The initial stress required to reach specified total first-creep deformations in 24 hours was obtained at various relative humidities from Figure 29. These data are summarized in Table XX.

TABLE XX

INITIAL STRESS VERSUS RELATIVE HUMIDITY AT VARIOUS
TOTAL 24-HOUR FIRST-CREEP DEFORMATIONS

Total 24-hour first-creep deformation, %	0.5	1.0	2.0	3.0
Initial stress at 23.5% R.H., kg./sq. mm.	3.15	4.60	5.85	- -
Initial stress at 50% R.H., kg./sq. mm.	2.50	3.60	4.85	5.85
Initial stress at 63% R.H., kg./sq. mm.	1.95	2.80	3.85	4.60
Initial stress at 73.5% R.H., kg./sq. mm.	1.60	2.35	3.30	4.0
Initial stress at 83% R.H., kg./sq. mm.	1.15	1.75	2.60	3.35
Initial stress at 94% R.H., kg./sq. mm.	0.60	0.95	1.65	2.25

It was found useful to plot these initial stress values versus 100 minus the relative humidity in per cent on a log-log plot. See Figure 31. Straight lines may be fitted to the points at the four highest relative humidities at all levels of deformation. The

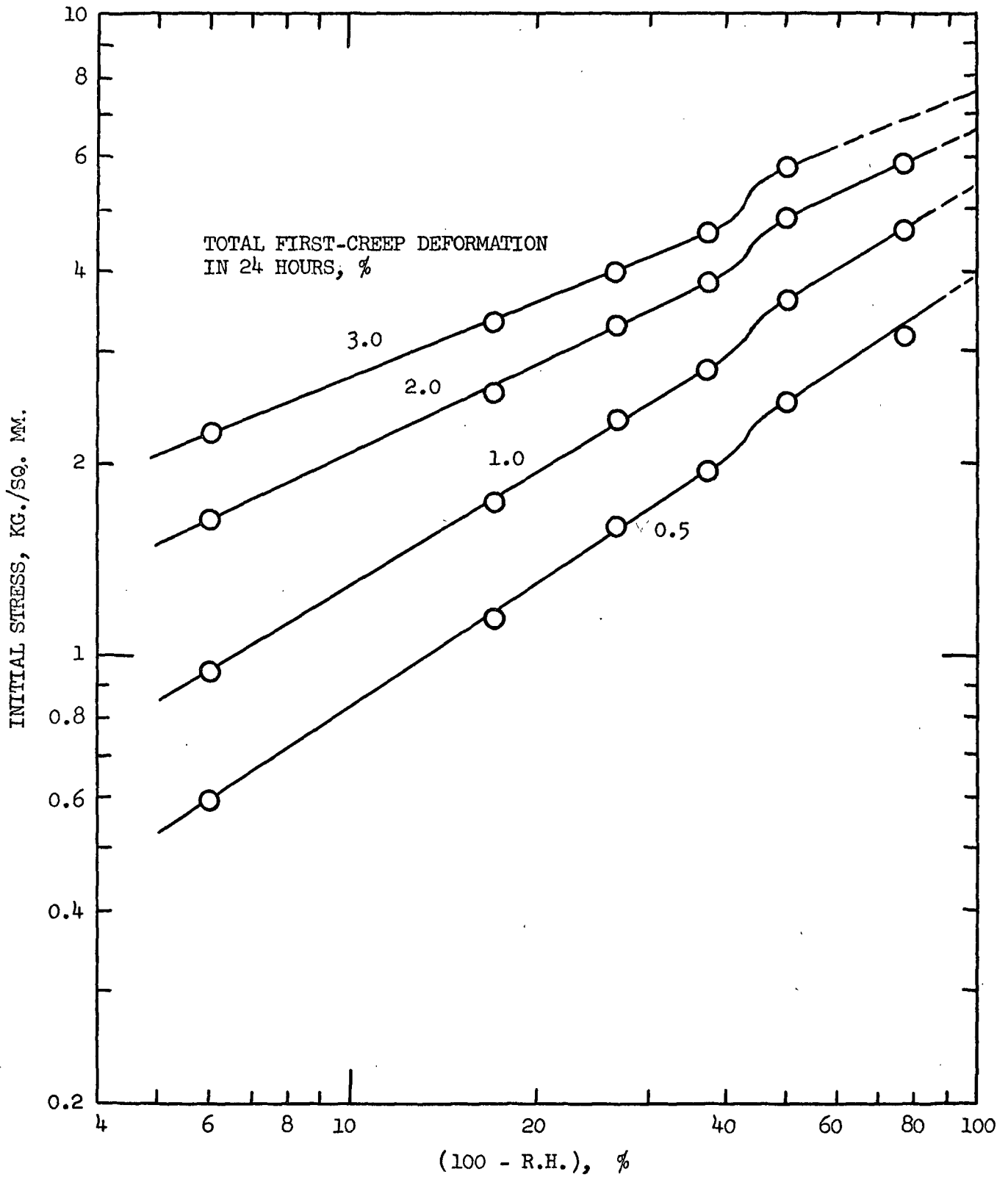


Fig. 31. Initial Stress Required to Reach Various Total First-Creep Deformations in 24 Hours Versus (100 - R.H.) in Per Cent

experimental points at 50 and 23.5% R.H. lie on lines which are approximately parallel to those at higher relative humidities, but are displaced upward by greater amounts at greater total first-creep deformations. A break similar to that shown in Figure 31 was marked for the relative delayed deformation, but was not detectable in similar plots for the total first-recovery deformation in 24 hours. The break must be ascribed to differences in the delayed first-creep deformation.

These rather abrupt breaks in response between 50 and 63% R.H. were analyzed in considerable detail. They occur as well in shorter test durations and could not be linked to either the short or long duration creep response. The straight-line relationships of Figure 31 will apply over wide ranges of test duration and total deformations. The breaks in response are much sharper when deformation is plotted versus moisture content, and will be noted in plots of the initial stress required to reach specified levels of total first-recovery deformation versus moisture content. The apparent elastic modulus versus relative humidity curve changes its form rather sharply at about 55% R.H. A more abrupt change occurs in plots of apparent elastic modulus versus moisture content.

Similar breaks in response versus relative humidity could be produced by partially mechanically conditioning a number of specimens followed by desorption and testing at various relative humidities on the adsorption curve. The breaks occur in this case because mechanical conditioning is effective at relative humidities below the value of the mechanical conditioning test, but the mechanically conditioned character of the specimens is progressively destroyed at higher relative humidities.

This subject is discussed more fully in the section relating to swelling recovery. It might be suggested, tentatively, that these specimens are in a partially mechanically conditioned state as a result of the planar restraint during drying and that this effect accounts for the break in response as a function of relative humidity on the adsorption curve. This possibility was tested and is discussed later in relation to the effect of humidification on creep behavior.

Similar breaks in response were reported by Andersson and Berklyto (8) in the load-deformation properties of machine-made papers as functions of relative humidity and moisture content. The breaks were greater for specimens in the machine direction and occurred between 60 and 70% R.H. in an adsorption series of tests. Rather sharp changes in the electrical resistance of paper have been reported at about 52% R.H. (64). It was suggested that these changes may have been due to modifications in the manner by which water is bound to cellulose. The abruptness of the break in the present work and the small range of moisture content over which it occurs suggest perhaps that it is due to an irreversible structural change, which occurs most rapidly in that small range of moisture content. Unfortunately, no data were available on the reversibility of this effect.

FIRST-RECOVERY RESPONSE

The total first-recovery deformation in 24 hours are plotted versus initial stress in Figure 32 at various relative humidities. The curves are slightly concave upward at lower relative humidities and approach linearity at higher relative humidities. The differences in shape of

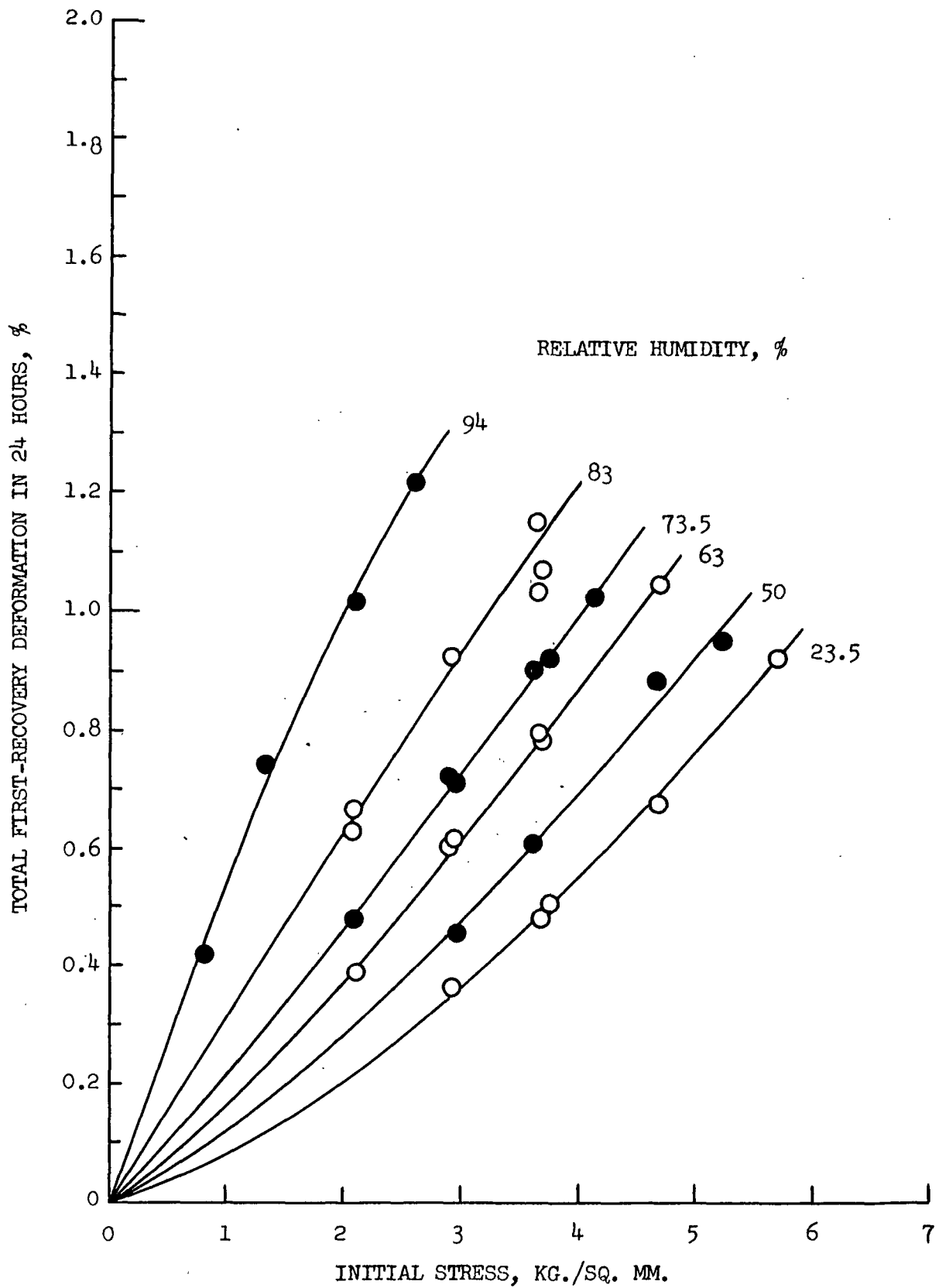


Fig. 32. Total First-Recovery Deformation Versus Initial Stress at Various Relative Humidities in 24-Hr. Tests

these curves can be traced to the fact that longer times of loading are required to reach the limiting value of recoverable deformation at the lower relative humidities, and that at constant test durations, insufficient time will have elapsed to approach the limiting value of recovery deformation at the lower initial stresses. The test duration may be long enough to reach those limiting values in the tests at higher stresses, and the recovery deformation versus initial stress curves in this range may be described by the upper portions of straight lines passing through the origin. This is expected from a study of earlier data summarized in Table IX and plotted in Figure 12. Hence, in tests of constant duration, the recovery deformation versus initial stress curve may actually be concave upward at low stresses and become asymptotic to a straight line passing through the origin at higher stresses. In the absence of sufficient data to demonstrate this expected curve shape, the experimental points were connected by the smoothest curves.

A limiting value of total recoverable deformation is expected at all relative humidities. This is demonstrated by the plots of the total 24-hour first-recovery strain per unit initial stress versus the total 24-hour first-creep strain per unit initial stress shown in Figure 33. The total recovery in 24 hours usually is over 90% of the value which would be obtained by extrapolation of the extended recovery curve to zero slope at long times. At 94% R.H., a maximum total extrapolated first-recovery strain per unit initial stress was reached between 1.0 and 1.5 kg./sq. mm. At 2.6 kg./sq. mm., the actual extrapolated total first-recovery deformation was about 15% lower than the maximum. This reduction appears comparable to the decrease in the short-time recovery

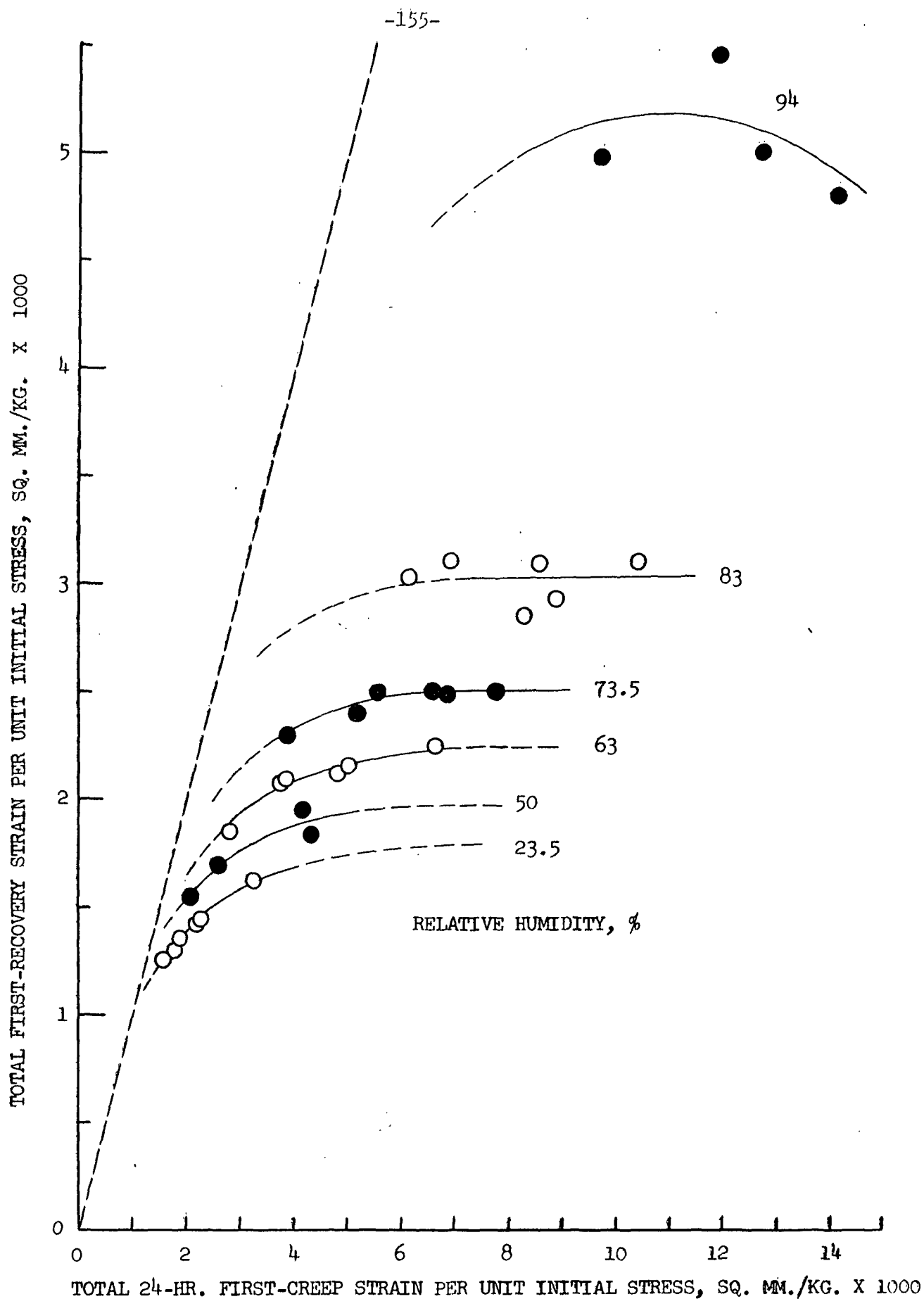


Fig. 33. Total First-Recovery Deformation Versus Total First-Creep Deformation in 24-Hour Tests at Various Relative Humidities

with greater time of loading in tests at constant initial stress at 50% R.H. (see Figure 16). It may be attributable to nonrecoverable deformation introduced in the logarithmic creep range. The mechanisms of logarithmic creep must account for changes in structure which are compatible with decreasing recovery both at early times and in total amount.

The first-recovery curves in these 24-hour tests were nearly linear at most initial stresses at all relative humidities. The chief differences between the recovery curves were the greater slopes at higher relative humidities when compared at equal levels of total first-recovery deformation. This was expected since it was shown earlier that the recovery curves of multiple-cycle tests at higher relative humidities were steeper than the preceding primary creep curves.

The relationships between the total first-recovery and the total first-creep deformations in 24-hour tests are shown as a function of relative humidity in Figure 34. In this plot, the total 24-hour first-creep deformations have been plotted versus relative humidity at several constant total first-recovery deformations in 24-hour tests. A high point on any curve represents a minimum percentage of recovery. It may be seen that the minimums in percentage of recovery at the higher deformations occur between 70 and 80% R.H. In other words, the greatest nonrecoverable deformations will occur in this range of relative humidity. At lower levels of deformation, the peaks do not appear because the immediate elastic deformation is a large percentage of the total deformation. The abrupt breaks in the curves at about 55% R.H. at the two lower first-recovery deformations are due to rather sudden increases in the

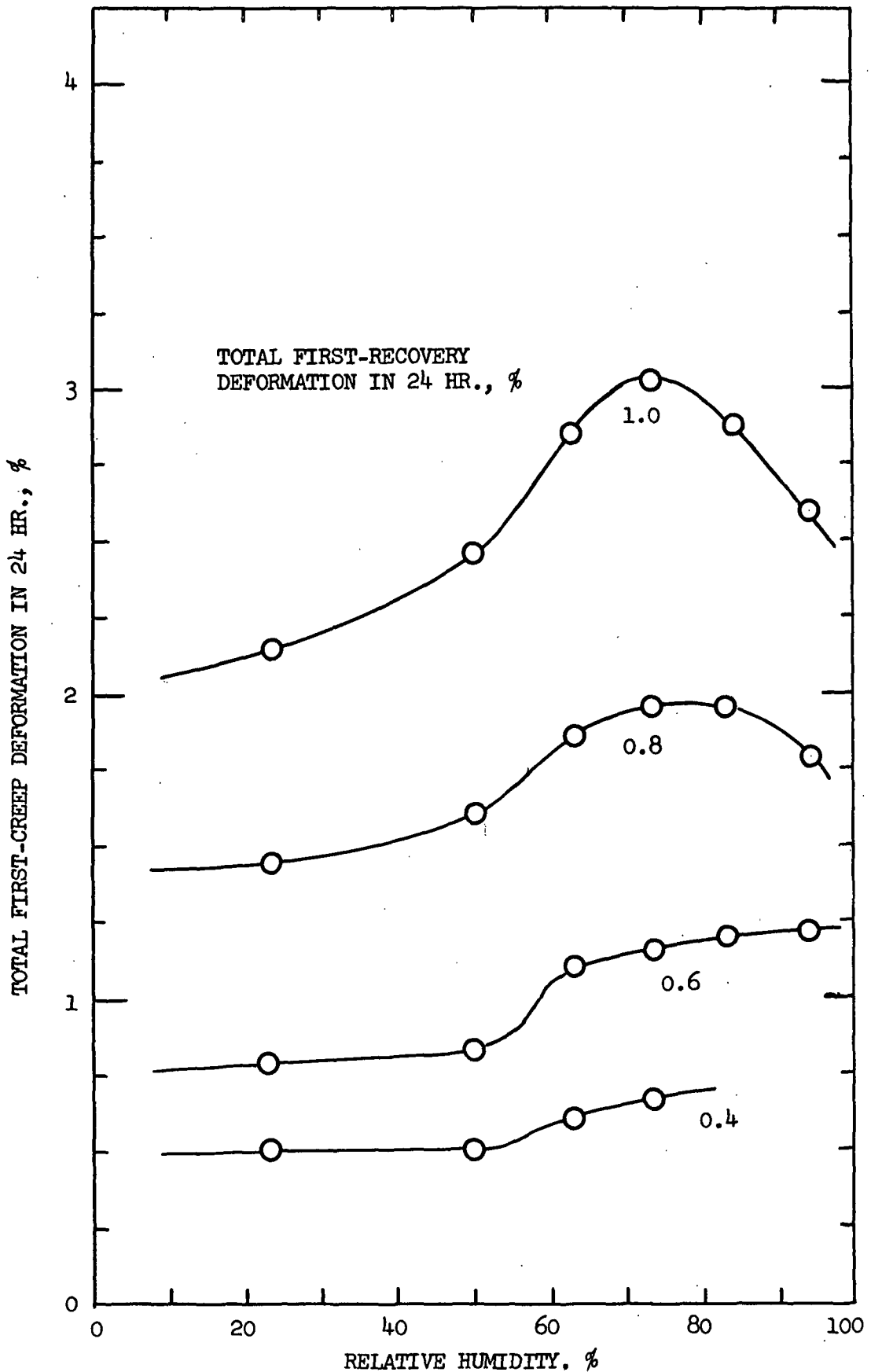


Fig. 34. Total First-Creep Deformation Versus Relative Humidity at Various Total First-Recovery Deformations in 24-Hr. Tests

delayed response and not to rapid changes in the apparent elastic modulus. If only delayed deformations were considered, the same minimums in percentage of recovery will occur between 70 and 80% R.H., but the recoverability at 94% R.H. becomes more pronounced. These effects defy simple explanation. On one hand, it might be expected that the percentage of recovery would increase with increasing relative humidity because of reduced intermolecular bonding and a tendency for less inhibition of recovery by the formation of metastable configurational structures. It also seems probable, that structural changes involving the crystalline portions of the polymer would occur more easily at higher relative humidities. This might tend to reduce the recoverability of given total first-creep deformations. Possibly, this behavior may be a combination of several effects, and must be interpreted in terms of the effect of relative humidity on two or more mechanisms of response.

THE EFFECT OF HUMIDIFICATION ON CREEP PROPERTIES

In the preceding section, it was shown that breaks occurred in curves relating factors of first-creep response to relative humidity. It was suggested that the low first-creep response at the lower relative humidities may be a consequence of the residual strain introduced during drying of these specimens, particularly, since similar breaks were not significant in first-recovery properties. As a general rule, it was noted that mechanical conditioning was effective at all relative humidities lower than the test humidity, but that the mechanically conditioned state was progressively destroyed at higher relative humidities. This latter effect is illustrated later in swelling recovery tests. The break in

response could be due to a partial mechanical conditioning of the specimen during drying. It is the purpose of the following tests to determine whether this may be the case by subjecting specimens to high relative humidities followed by desorption and testing at various lower relative humidities.

Six specimens of Handsheet 29 were inserted in creep testing units at 50% R.H. in the as-dried condition to an initial length of 10.00 inches. The relative humidity was increased to about 97% in the humidification treatment and maintained there for 48 hours. The oven-dry moisture content was about 24%. The relative humidity was then decreased to 83%, and one of the specimens was subjected to a first-creep test at an initial stress of 3.75 kg./sq. mm. The specimen broke in this test and the 24-hour total first-creep deformation was obtained by extrapolation. Following this test, the relative humidity was decreased to 73.5% and 24-hour first-creep and first-recovery tests were run on a second specimen at the same initial stress. Other specimens were tested in similar tests at 63, 50, and 23.5% R.H., where each relative humidity was reached in sequence by desorption.

A summary of these tests is given in Table XXI. The total first-creep and first-recovery deformations in the adsorption series of tests at the same initial stress were obtained from Figures 29, 30, and 32. These data are summarized in Table XXII. A comparison of the creep and recovery response in first tests before and after humidification is shown in Figure 35 as a function of relative humidity. The moisture contents of the specimens differ due to adsorption-desorption hysteresis.

TABLE XXI

TESTS OF HUMIDIFIED SPECIMENS

Relative Humidity of Test Reached by
Desorption from 97% R.H.

Creep Load, 3.50 kg.

Test number	97	99	100	101	98	102
Specimen number	29-7	29-6	29-5	29-10	29-8	29-9
Relative humidity, %	83	73.5	63	50	50	23.5
Moisture content, % (ovendry)	16.1	12.6	10.4	8.0	8.0	4.6
Initial stress, kg./sq. mm.	3.74	3.74	3.74	3.76	3.76	3.76
Total first-creep deformation in 24 hours, %	3.63 ^a	2.72	2.13	1.72	1.61	0.93
Total first-recovery deformation in 24 hours, %	- -	1.02	0.88	0.77	0.74	0.56
Relative delayed deformation, %	1.35 ^a	1.23	1.12	0.98	0.91	0.46

^a Obtained by extrapolation of first-creep curve.

TABLE XXII

DEFORMATIONS IN CREEP AND RECOVERY AT 3.75 KG./SQ. MM.
VERSUS RELATIVE HUMIDITY BEFORE HUMIDIFICATION

Relative Humidity, %	Total First-Creep Deformation in 24 Hr., %	Relative Delayed Deformation, %	Total First-Recovery Deformation in 24 Hr., %
23.5	0.68	0.29	0.50
50	1.09	0.62	0.64
63	1.88	1.17	0.81
73.5	2.63	1.47	0.93
83	3.65	1.65	1.15

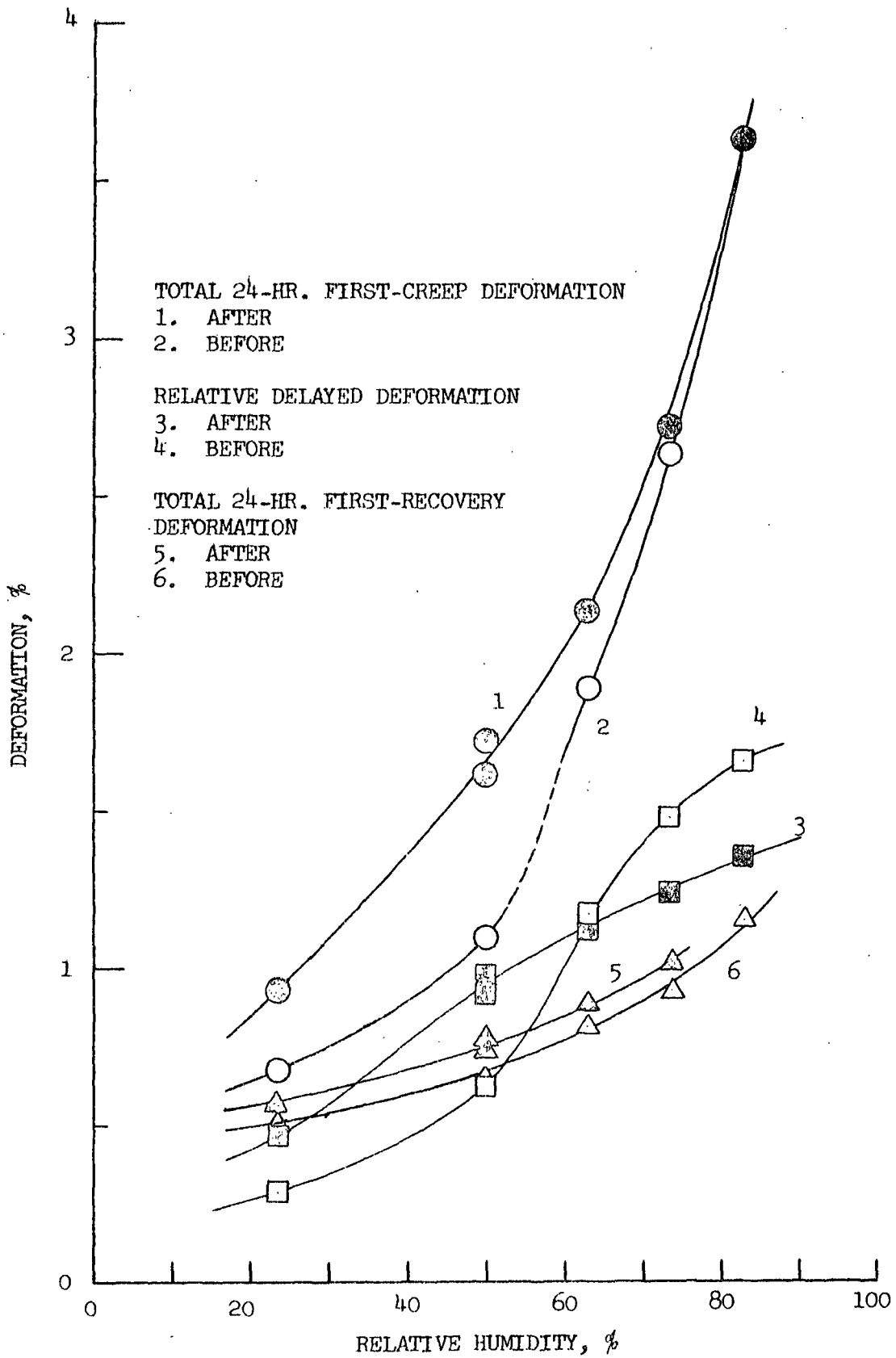


Fig. 35. Comparison of Creep Properties at 3.75 kg./sq. mm.
Before and After Humidification

The use of moisture contents in place of relative humidity as an independent variable would alter the details of the comparison, but not the effects on which the following discussion is based.

After humidification, the total first-creep deformation in 24 hours was increased most at 50 and 23.5% R.H., and insignificantly at relative humidities above about 75%. The break in response was not evident in the tests of humidified specimens. It must be concluded that the break in creep response versus relative humidity in tests on the adsorption curve represents a lack of response at the lower relative humidities due to structural changes, apparently induced as a result of drying under external planar restraint. It cannot be stated whether the abrupt rise in response at about 55% in the adsorption tests occurs at that point because these specimens were dried under restraint to that relative humidity, or whether changes in the mechanisms of cellulose-water binding at that relative humidity provide for an initially large change in structure.

The total first-recovery deformation was increased slightly at all relative humidities. Plots of the total first-recovery deformation versus moisture content for the adsorption series of tests showed slight breaks in the curve between 7 and 8% moisture content. These were not apparent after humidification. It must be pointed out that these data do not prove conclusively that the break in response versus moisture content will not occur after humidification. It is entirely possible that the break in response has merely been shifted to lower relative humidities. Unfortunately, data were not obtained at relative humidities in the range

of 30 to 40% which would test that possibility. If it would occur, however, the break would be much smaller in magnitude. The author feels that the break will not occur to a significant degree after humidification. It is felt that humidification would have only a small effect on the creep behavior at 0% R.H. in tests at constant initial stress, and that the curves drawn in Figure 35 are good approximations to the actual relationships.

The humidification treatment resulted in differences in the shapes of the first-creep curves. This is illustrated by the differences in the two curves relating the relative delayed deformations between 10 seconds and 24 hours (86,400 seconds) to relative humidity. Before humidification, the relative delayed deformation increased rapidly between 50 and 60% R.H. The rapid rise in relative delayed deformation did not occur after humidification. The shape of the curve for the humidified specimens is in doubt at the lower relative humidities. Thus, although the total 24-hour first-creep deformation was not altered appreciably by humidification, a greater percentage of that deformation occurred before 10 seconds. The percentage recovery in 24-hour tests did not change very much, and a comparison of the recovery curves indicated that the increase in delayed recoverable deformation was distributed uniformly in time. The apparent elastic modulus decreased as a result of humidification at all relative humidities, but it was not possible to obtain accurate estimates of the magnitude of the decrease. The changes in the relative delayed deformation versus relative humidity curves, therefore, are largely changes in the time distribution of

the nonrecoverable deformation. The change in distribution occurs principally at the higher relative humidities, since at 50 and 23.5% R.H. only slight changes in the time distribution of the total first-creep deformation occurred as a result of humidification.

The slopes of the logarithmic creep portions of the first-creep curves of humidified specimens (Figure 36) are the same between 50 and 83% R.H., and possibly would be similar at 23.5% R.H. At long times, the creep rate at a given time is not a function of relative humidity. This is the first clear indication that the mechanisms of response of the logarithmic creep range may be basically independent of moisture content. This did not occur in tests before humidification. The slopes of the logarithmic creep portions of first-creep curves before and after humidification are summarized in Table XXIII. It has been shown that the slopes are linearly related to initial stress below about 70% R.H., but were less dependent on initial stress at higher relative humidities. The results of the creep and swelling recovery tests, described later, indicated that a linear relationship between the constant K of the logarithmic creep equation and initial stress would apply as well after humidification, at least in tests at 50% R.H. The K/S_0 values are plotted versus relative humidity in Figure 37. The curves must be regarded as specific to an initial stress of 3.75 kg./sq. mm.

The effect of humidification on the creep property-relative humidity relationships stresses the importance of interpreting those relationships in terms of the conditions of specimen preparation and the

TABLE XXIII
SLOPES OF LOGARITHMIC-CREEP PORTIONS OF FIRST-CREEP CURVES
VERSUS RELATIVE HUMIDITY

Test Number	Calculated Initial Stress, S_0 , kg./sq. mm.	Relative Humidity, %	Slope, %/Decade of log time	K/S_0 , sq. mm./kg. x 10,000
Before Humidification				
71	5.69	23.5	0.337	5.93
133	4.66	50	0.337	7.23
135	5.22	50	0.370	7.09
52	3.68	63	0.320	8.70
55	4.67	63	0.420	8.99
85	3.61	73.5	0.360	9.97
81	3.75	73.5	0.360	9.60
80	4.12	73.5	0.393	9.54
45	3.62	83	0.350	9.67
41	3.68	83	0.373	10.14
40	3.65	83	0.373	10.22
127	2.09	94	0.333	15.93
128	2.60	94	0.360	13.85
After Humidification				
98	3.76	50	0.282	7.50
101	3.76	50	0.288	7.66
100	3.74	63	0.288	7.70
99	3.74	73.5	0.290	7.76
97	3.74	83	0.289	7.73

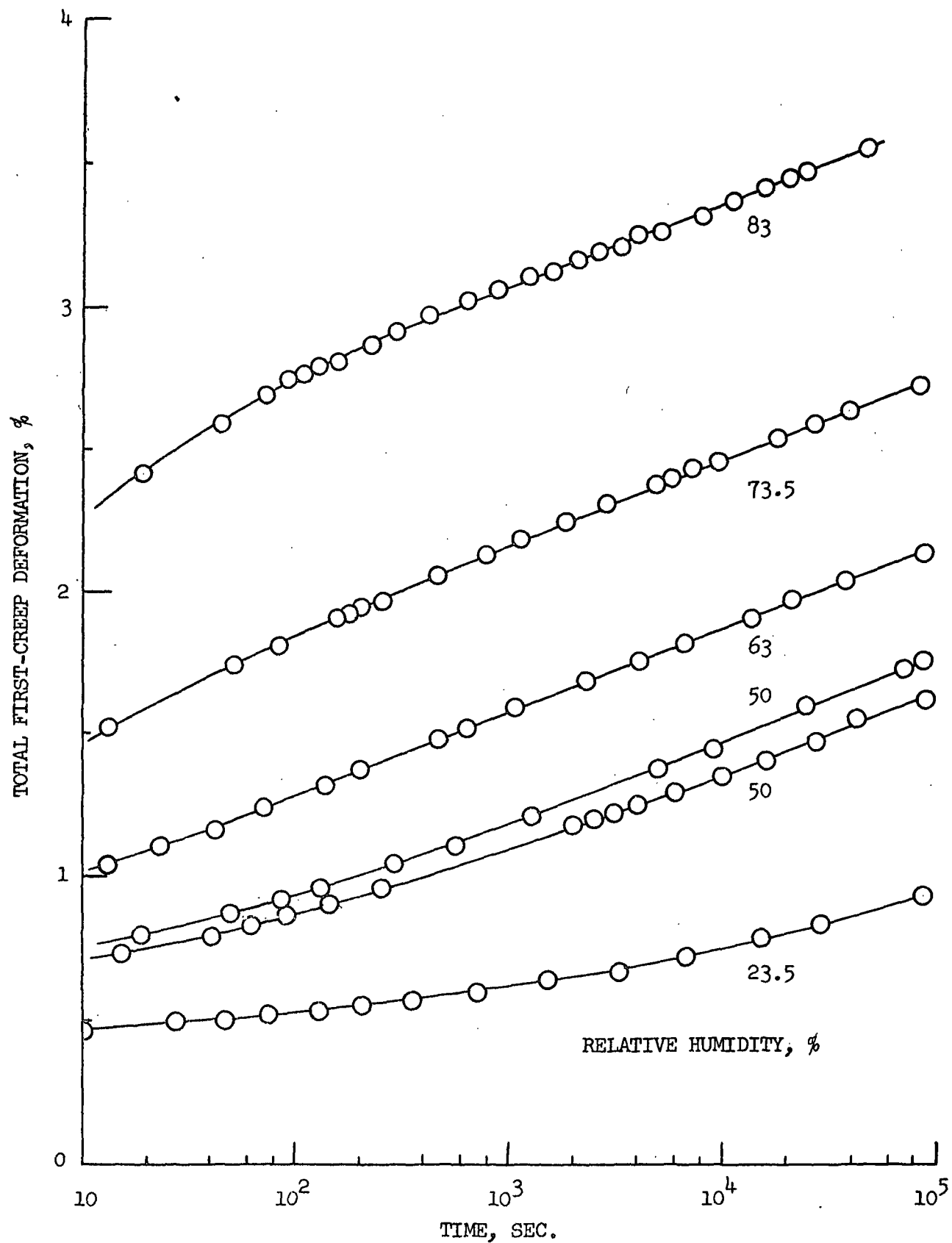


Fig. 36. First-Creep Curves of Humidified Specimens at 3.75 kg./sq. mm.

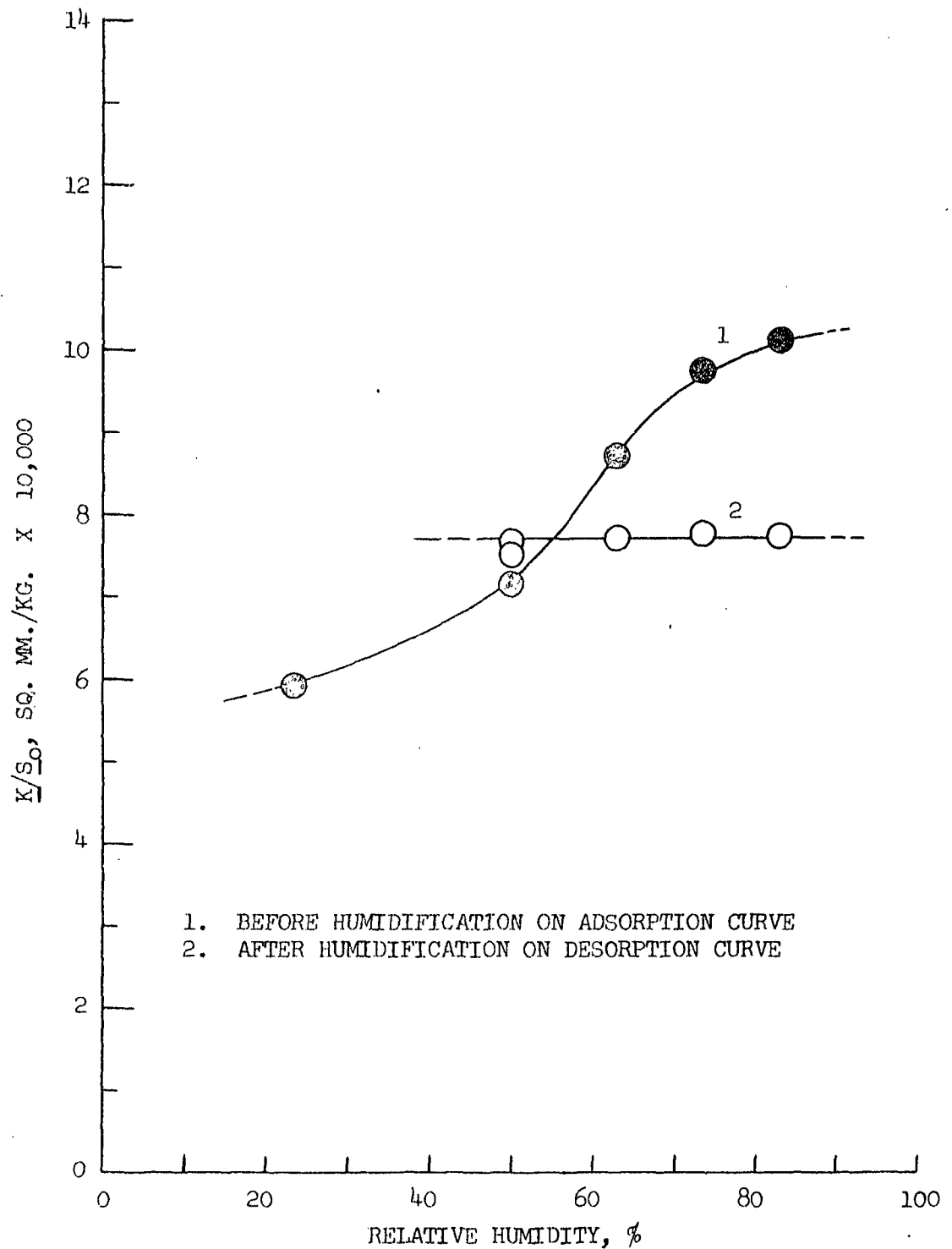


Fig. 37. Relationship Between $\frac{K}{S_0}$ and Relative Humidity in First-Creep Tests at 3.75 kg./sq. mm. Before and After Humidification

previous moisture content history of the specimen. Any interpretation of the fact that the rates of creep are the same at given long times in the first tests of humidified specimens over rather wide ranges of relative humidity must account also for the variations that occur with relative humidity before humidification. It is highly unlikely that mechanisms of response of configurational elasticity or fiber-fiber bond ruptures could account for this behavior, since, essentially, this would require that changes in moisture content from at least 8 to 16% had no influence on the retardation times or on the strengths of the fiber-fiber bonds. The most logical explanation of this behavior involves changes in the crystalline structure, probably by increases in crystallinity. It might also be suggested that the greater deformations prior to logarithmic creep at higher relative humidities could act to reduce the rate of logarithmic creep and exactly counterbalance the rate-increasing tendency of the increased moisture content. This seems unlikely, however, since such behavior was not evident in tests before humidification.

Stress versus crystallization-rate relationships could be exceedingly complex in cellulose and strongly dependent on both relative humidity and initial molecular structure. The evidence supporting crystallization as the principal mechanism of response of the longer duration logarithmic creep deformation is entirely indirect in the sense that mechanical behavior alone does not constitute direct proof of crystallite growth. The suggestion that crystallite growth is a mechanism of response in the creep of paper is hypothetical, and the results of this study are not a test of that hypothesis.

The crystallization of high polymers under stress is an exceedingly complex subject. The writer has not found, in existing literature, any explanations of high-polymer crystallization, particularly in terms of rate, that might be applicable in this study. Unfortunately, the creep data of many polymers are essentially rate data, and equilibrium states are seldom reached within reasonable experimental time intervals. It seems likely that the complexity of crystallization phenomena in high polymers may be related to the restraint of those segments of molecules which are in position to rotate into parallelism and form a part of the crystalline structure by adjacent portions of the molecule in the amorphous regions. Crystallization rate, therefore, could easily be dependent on the rate of continued configurational change in the fringe or amorphous areas of the polymer. This seems most plausible at the low relative humidities, where the intermolecular bonding forces will be greatest. Less restraint by the amorphous polymer might be noted at high relative humidities, which would result in easier crystallization. In any case, the driving force promoting crystallization is probably related most closely to the configurational structure of the stretched polymer, and, therefore, to the initial molecular configuration and the changes which occurred with deformation.

Added support for crystallization as a mechanism of the longer duration logarithmic response in creep tests would be provided, by elimination of other mechanisms from consideration, if it could be established that the logarithmic response at high relative humidities and the higher initial stresses was identical (same creep rate versus time relationship) for handsheets of different degrees of intermolecular bonding. Work along

this line was discontinued before this effect could be established, although the crude preliminary results indicated that such behavior might occur at 94% R.H.

A study of the creep properties of handsheets of different solid fraction at various relative humidities before and after humidification would be useful in further definition of the mechanisms of response in creep tests.

SWELLING RECOVERY

Substantial portions of the first-creep deformation of paper are nonrecoverable under the test conditions of temperature and relative humidity. The sigmoidal recovery curve indicates that the nonrecoverable deformation must be considered permanent at the test conditions. A measure of the true permanence of the nonrecoverable deformation may be obtained by changing the external conditions to promote swelling and plasticization of the polymer. If these deformations are recoverable by swelling treatments, it must be assumed that the nonrecovery was due to the formation of a metastable molecular structure. Truly permanent deformations will not be recoverable by swelling treatments. Swelling reduces the intermolecular bonding and allows the inhibited recovery to proceed at measurable rates to a new condition of structural equilibrium. Swelling recovery of all or part of the nonrecoverable deformation of viscose (3, 13), cellulose acetate (3, 38), silk (2), wool (65), etc., have been demonstrated either by increases in temperature or wetting of the polymer.

Experimentally, it would be possible to assess the true permanence of nonrecoverable deformation by swelling the polymer to the same degree which existed during its preparation. In paper and possibly in other polymers this may not be possible, since it is unlikely that the specimen could be swollen to the necessary degree without additional mechanical treatment. Thus, it is necessary to subject the specimen to arbitrary swelling treatments and determine the relative recoverability.

Additional treatments and more severe swelling conditions may increase the amount of swelling recovery. That portion which is recovered by swelling must be attributed to the destruction of a metastable molecular structure, but the deformation resistant to recovery by swelling is not necessarily permanent.

A series of creep, creep recovery, and swelling recovery tests were run on Specimen 55-8. The specimen was inserted in the clamps in the as-dried condition at 50% R.H. It was immediately humidified at 97.8% R.H. for 12 hours while within clamps (over a saturated solution of potassium sulfate). The approximate moisture content was 24% on the oven-dry basis. The temperature in the humidification and mechanical tests was maintained between 72 and 74°F. After redrying at 50% R.H. and no load, a net contraction in length of 0.48% was observed. The specimen was loaded to 3.5 kg./sq. mm. in a creep test of 24-hour duration. The total creep deformation was 1.13% and the total recovery deformation in 24 hours was 0.62%. Total first-creep deformations of 0.65 and 0.73% in 24 hours and total first-recovery deformations of 0.48 and 0.53% in 24 hours were obtained in Tests 78 and 90 at the same initial stress on specimens of the same sheet before humidification. The increase in total first-creep deformation was of the same order of magnitude as the net contraction due to humidification. About half of the increased recovery deformation was due to an increase in immediate elastic deformation. The apparent elastic modulus decreased from about 1150 to 900 kg./sq. mm. as a result of humidification. A second cycle of creep and recovery was run on this specimen, and the second-recovery period

was extended to 169 hours. At the end of this period, the nonrecoverable deformation totaled 0.54%. The specimen was rehumidified at 97.8% R.H. for 24 hours and reconditioned at 50% R.H., which reduced the nonrecoverable deformation to 0.08%. A third-creep test at the same initial stress yielded a total 24-hour creep deformation of 1.11%. At the end of a 171-hour recovery period, the nonrecoverable deformation was 0.43%, which was recovered completely in another humidification cycle. These and the following tests on this specimen are summarized in Table XXIV.

At 4.5 kg./sq. mm., the total creep deformation in 24 hours was 1.58% in a test on the same specimen following humidification. The total recovery deformation in 120 hours was 0.88%. Humidification reduced the nonrecoverable deformation from 0.70 to 0.07%. The total first-creep deformation in 24-hours on an as-dried specimen of Handsheet 55 at 4.48 kg./sq. mm. was 1.23% (Test 78). The specimen broke in the test at 5.5 kg./sq. mm.; however, the extrapolated total creep deformation equaled 2.41% compared to 2.03% in Test 79 on an as-dried specimen at about the same stress. The increase in total creep deformation is in the same order of magnitude as the contraction due to humidification. This condition may be realized only if the creep test is continued approximately to the onset of logarithmic creep.

An estimate of the swelling recovery at 5.45 kg./sq. mm. was obtained in Test 79. After 5 cycles of 24-hour duration creep and recovery tests and an extended fifth-recovery period at 50% R.H., the nonrecoverable deformation was 1.41%. The specimen was humidified for 4 hours over a 5% sulfuric acid solution at an estimated relative humidity of

TABLE XXIV

SUMMARY OF CREEP AND SWELLING RECOVERY IN TEST 89

Specimen 55-8, Specimen Dimensions, 1 x 10 inches
Relative Humidity, 50% in Creep and Recovery Tests
Temperature, 72 to 74°F.

Description	Stress, kg./sq. mm.	Relative Humidity, %	Time of Test, hr.	Micrometer Reading at End of Test, in.	Total Deformation in Creep or Recovery Test, %
Initial specimen	0	50	- -	0.8274	- -
Humidification	0	97.8	12	- -	- -
Conditioning	0	50	97	0.8755	- -
Creep	3.5	50	24	0.7630	1.13
Recovery	0	50	24	0.8248	0.62
Creep	3.5	50	24	0.7588	0.66
Recovery	0	50	24	0.8161	0.57
Additional recovery	0	50	145	0.8211	- -
Humidification	0	97.8	12	- -	- -
Conditioning	0	50	48	0.8675	0.08 ^a
Creep (Test 89-A)	3.5	50	24	0.7569	1.11
Recovery	0	- -	24	0.8207	0.64
Additional recovery	0	- -	147	0.8245	- -
Conditioning	0	11.1	19	- -	- -
Humidification	0	97.8	26	- -	- -
Conditioning	0	50	23	0.8683	0.01 ^a
Creep	3.5	- -	24	0.7517	1.17
Recovery	0	- -	24	0.8133	0.62
Additional recovery	0	- -	143	0.8196	- -
Humidification	0	97.8	24	- -	- -
Conditioning	0	50	260	0.8658	0.03 ^a
Creep	3.5	50	24	0.7644	1.01
Recovery	0	50	24	0.8266	0.62
Creep	4.5	50	24	0.7120	1.14
Recovery	0	50	24	0.7945	0.83
Additional recovery	0	50	263	0.8027	- -
Humidification	0	97.8	24	- -	- -
Conditioning	0	50	383	0.8575	0.08 ^a
Creep (Test 89-B)	4.5	50	24	0.6993	1.58
Recovery	0	50	24	0.7824	0.83
Additional recovery	0	50	96	0.7877	- -
Humidification	0	97.8	24	- -	- -
Conditioning	0	50	96	0.8504	0.07 ^a
Creep (Test 89-C)	5.5	50	--	rupture	- -

^a Nonrecoverable deformation relative to specimen length after previous humidification cycle.

97% (66). The nonrecoverable deformation was reduced to 0.25%, but part of the recovery may be due to recovery of residual strain. Following two additional cycles of 24-hour tests at 5.45 kg./sq. mm. and an extended recovery period, the nonrecoverable deformation was 1.28% and the maximum creep deformation was 2.27%. Another humidification treatment over 5% sulfuric acid for 4 hours reduced the nonrecoverable deformation to 0.41%. The total recovery in these latter tests was 1.86% with humidification compared to 0.99% at the test conditions. The important consideration is that, at this higher stress, some of the deformation was nonrecoverable by humidification at 98% R.H. Over the entire series of tests in Test 89, the total nonrecoverable deformation after humidification was 0.25%. Thus, only a small fraction of the creep deformation at 3.5 and 4.5 kg./sq. mm. is resistant to recovery at the arbitrary humidification conditions. The total recoverable strain per unit initial stress with humidification is about 3.4×10^{-3} sq. mm./kg. for stresses of 4.5 and 5.5 kg./sq. mm. Logarithmic creep begins at strain per unit stress values of 2.5 to 2.8×10^{-3} sq. mm./kg., hence, some of the swelling recovery includes the nonrecoverable logarithmic creep deformation.

A number of spot checks throughout the course of this work indicate that the following effects may be expected.

1. Complete wetting will increase the swelling recovery beyond that which can be obtained at 98% R.H. and humidification at lower relative humidities is less effective. Swelling recovery can also be obtained in part by wetting with acetone.

2. The extent of recovery by swelling, particularly for nonrecoverable deformations introduced at relative humidities above 70%, may reach a maximum and decrease with increasing nonrecoverable deformation.

It is self-evident that nonrecoverable deformations introduced at high relative humidities will be less recoverable by humidification. Swelling recovery is possible when the nonrecoverable deformation is introduced at relative humidities where the formation of a metastable structure may occur. It cannot be stated whether the deformation recoverable by swelling is due to inhibited configurational elasticity or other effects.

A comparison of the creep curves after humidification at different stresses in tests on a single specimen is difficult, but apparently it will be possible to form a master creep curve for humidified specimens in the manner employed for Handsheet 23.

Since the total first-creep deformation at any stress is increased approximately by the amount of the contraction due to humidification, the time shift per unit stress in forming a master creep curve will be reduced. This is contrary to an earlier suggestion that the time-shift requirement may be related to the degree of molecular order. Humidification, however, may prove to be a special condition in influencing molecular structure. The effect of humidification on creep behavior is complex and often contrary to the effects of beating and wet pressing. A study of the effect of humidification on specimens of varying sheet structure would help clarify these effects. In most respects, the recovery of residual strain introduced during drying and the recovery of nonrecoverable deformations of creep tests are comparable.

HYGROEXPANSIVITY

The same structural factors which influence the mechanical properties of paper are likely to influence the hygroexpansivity of the handsheets, though possibly, in different ways. The hygroexpansivity of various specimens of these handsheets was determined to provide estimates of the dimensional changes which occurred prior to testing at different relative humidities and to investigate possible correlations between mechanical behavior and hygroexpansivity.

Twenty specimens were inserted in the Neenah Expansimeter at 50% R.H. in the as-dried condition. Each specimen was 1-inch wide and 10 inches in length. The testing procedures of Institute Method 539 were followed. The relative humidity cycle involved a preliminary conditioning at 50.9% R.H., followed by desorption to 12, adsorption to 89.8, and desorption to 50.9% R.H. The temperature varied between 73 and 77°F. during the course of the tests. A summary of the results is presented in Table XXV. These data represent changes in length relative to the initial length at 50.9% R.H. as percentages of the initial length. Negative values are contractions in length. A typical plot of the dimensional changes versus relative humidity is shown in Figure 38 (curve ABCD). In these hygroexpansivity tests, adsorption above 50% R.H. marks the first time the specimens were exposed to these higher relative humidities since their preparation. The measured extension in the first adsorption test is low at relative humidities above 50% due to progressive recovery of residual strain as the relative humidity is increased. The desorption curve from 89.8 to 50.9% R.H. seems to be of the proper shape

TABLE XXV

HYGROEXPANSIVITY OF ALPHA PULP HANDSHEETS

Temperature, 23 to 25°C.
Nominal Initial Specimen Length at 50.9% R.H., 10 inches
Relative Humidity Cycle, 50.9 to 12 to 89.8 to 50.9% R.H.

Specimen Number	36.5% R.H.	12.0% R.H.	36.4% R.H.	49.8% R.H.	69.3% R.H.	78.8% R.H.	89.8% R.H.	71.0% R.H.	50.9% R.H.
<u>Per Cent of Initial Length x 100</u>									
22-1	-9.2	-32.3	-16.9	-7.1	4.8	13.2	26.8	-0.3	-19.1
25-7	-11.2	-36.3	-19.5	-8.5	4.5	13.5	29.0	-2.3	-21.4
26-3	-11.0	-34.4	-19.1	-9.4	2.2	9.1	20.2	-6.0	-24.1
30-2	-11.1	-34.1	-19.2	-9.3	2.2	9.8	- -	- -	- -
33-5	-11.4	-37.1	-20.0	-8.8	4.7	14.3	30.9	-1.6	-23.2
34-10	-10.4	-35.6	-16.2	-5.9	5.1	12.9	25.8	-2.4	-24.5
36-6	-12.4	-38.3	-20.5	-9.1	4.3	13.4	27.8	-1.9	-22.5
39-3	-11.2	-35.2	-18.7	-8.5	3.7	11.9	24.7	-2.5	-21.8
41-9	-12.1	-37.7	-20.7	-9.3	5.0	15.6	33.6	2.8	-18.7
44-1	-12.2	-37.4	-20.3	-10.2	2.7	11.4	25.5	-4.3	-24.7
44-10	-13.0	-40.2	-21.9	-9.8	5.4	16.5	35.6	1.3	-21.9
46-9	-10.1	-29.8	-16.6	-8.1	1.8	8.9	20.7	-2.6	-18.0
48-9	-12.1	-35.4	-19.0	-10.2	0.1	6.5	17.2	-6.0	-22.7
50-10	-11.7	-35.0	-20.0	-10.2	0.8	9.3	20.2	-6.7	-25.9
52-11	-8.9	-26.6	-15.1	-7.5	1.1	7.2	17.1	-3.0	-16.8
53-10	-7.6	-25.8	-14.0	-7.4	1.6	7.3	16.3	-3.6	-17.3
55-1	-14.5	-41.8	-24.8	-13.6	-0.8	7.6	21.5	-11.4	-34.0
55-2	-14.0	-41.3	-24.1	-12.8	0.2	9.1	23.0	-9.6	-31.8
56-5	-13.0	-39.1	-21.2	-11.4	1.1	9.3	23.8	-9.9	-32.0
56-6	-11.8	-34.8	-20.1	-11.0	0.3	7.9	20.5	-8.3	-27.2

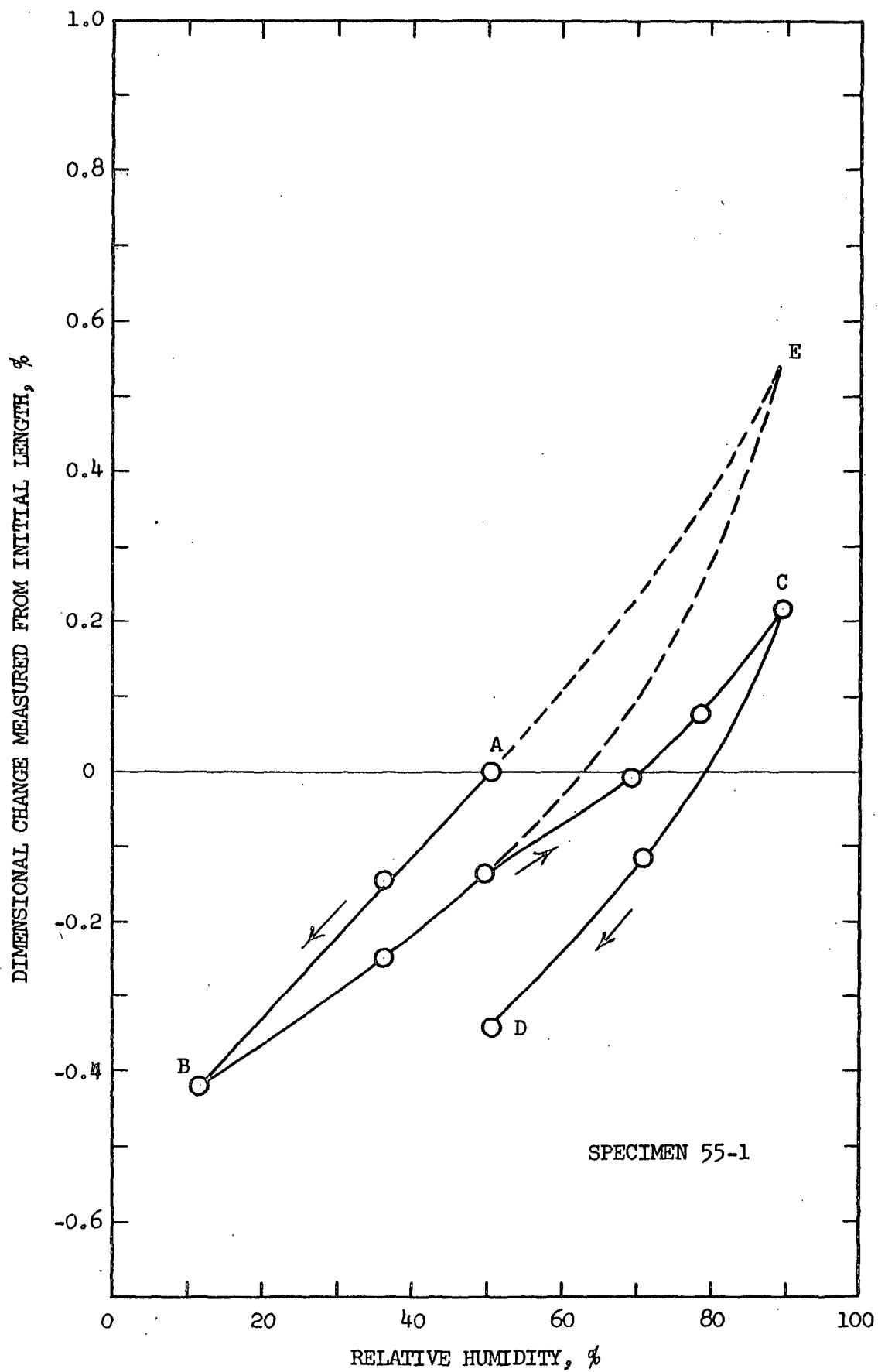


Fig. 38. Hygroexpansivity of Alpha Pulp Handsheets

to fit the desorption curve from 50.9 to 12% R.H., but is displaced downward by the amount of net contraction in the complete cycle. If the specimens were without residual strain (which might be the case after several adsorption-desorption cycles) the hygroexpansivity curve for a complete cycle should describe a closed hysteresis loop as shown by ABEA in Figure 38.

Varying amounts of net contraction were observed in hygroexpansivity tests by Larocque (67) for machine-made papers in first cycles. His desorption curves crossed and fell below the adsorption curve at higher relative humidities when the contractions were greater. The present data indicate the maximum in this type of effect, where the desorption curve falls below the adsorption curve immediately without any positive hysteresis.

The contractions in traversing the complete cycle were greatest for the handsheets prepared from 425 cc. S.-R. freeness pulp, were least for the on-20-mesh fraction of 775 cc. S.-R. freeness pulp, and were in the same order of magnitude for all other handsheets. This correlated generally with the percentage recovery in 24-hour first-creep and first-recovery tests (see Figure 22). The specimens with the greatest residual strain show the highest percentage recoveries. Similar behavior occurs if a specimen is partially mechanically conditioned, hence, it would appear that the residual strain and the nonrecoverable deformation of creep tests are due to the same type of mechanisms. It seems reasonable that if all of the specimens were humidified before testing, the percentage recoveries would be more nearly in the same order of magnitude for all handsheets.

The contraction during desorption generally follows the order indicated by the net recovery in the complete cycle. These data are too erratic to enable correlation of hygroexpansivity with creep properties; however, the specimens of greater creep response tend to exhibit lower hygroexpansivities. The exception is the handsheets prepared from screened pulp, in which the hygroexpansivity drops off sharply, and the creep response does not become greater. Weidner (68) has reported dimensional changes in the order of 1% in length and 8% in diameter for wood pulp fibers between 0 and 95% R.H. The dimensional changes in length of the specimens in this work are in the order of 1% over this range of relative humidity, which are comparable to Weidner's reported values in the length dimension.

It was noted that throughout the course of this work that if the relative humidity was decreased near the end of a creep test, while the specimen is under tensile load and the creep rate is small, smaller contractions in length occurred than for unloaded specimens. The net contractions may be very small at the higher tensile loads. If the load is removed at the lower relative humidity, creep recovery is not increased appreciably above values expected in tests at the lower relative humidity, which suggests that changes in molecular structure occurred. Similar effects might be expected to occur in the solid fibrous material adjacent to fiber-fiber bonds. Fibers in contact at angles to each other would be subjected to stresses with changing relative humidity as a result of differences in the shrinkage or expansion tendency in and across the fiber length dimension. The net changes in dimension with changing

relative humidity would be a compromise between the existing stresses and the relative ease with which deformation can occur in the principal fiber dimensions.

In view of the creep behavior and the effect of tensile load on hygroexpansivity, the writer feels that the relationship between hygroexpansivity and sheet structure in paper should be considered predominantly in terms of a heterogeneously restrained system. A greater amount of interfiber bonding, therefore, may increase the amount of solid fibrous material which is subjected to restraint preventing normal shrinkage and expansion with changing relative humidity. If it is assumed that the fibers are least deformable in their length dimension, the hygroexpansivity of paper should approach from higher values the hygroexpansivity of fibers in their length dimension.

SUMMARY AND CONCLUSIONS

This study deals with the tensile creep properties of handsheets prepared from softwood alpha pulp. All handsheets were dried on plates with essentially zero shrinkage in the plane of the sheet. Creep and creep recovery properties were investigated in greatest detail at 50% R.H. The preliminary work was followed by investigations of the effect of sheet structural variables and relative humidity on creep behavior. The general patterns of creep and creep recovery behavior are summarized here and a general interpretation of these results is presented.

In general, the response of paper in first-creep or in subsequent creep tests was very broadly distributed in time and may span 10 or more decades of log time. Unless otherwise indicated, the following behavior pertains to tests at 50% R.H. and 73°F. on specimens dried to equilibrium at those conditions.

The earlier response in first-creep tests at lower initial stresses could be fitted to exponential equations relating deformation to the time of loading. At longer times, exponential creep was followed by a transitional zone of creep in which the creep rate declined more rapidly with time than in the exponential creep zone. The transitional zone may span two or more decades of log time. Following the transitional zone of creep, the deformation was proportional to the logarithm of time (logarithmic creep). At intermediate initial stresses, all three zones of creep occur within an experimental time interval of 10 to 10^6 seconds. At low initial stresses, creep is largely exponential, whereas at higher initial stresses creep may be entirely logarithmic within this experimental

time interval. No departure from logarithmic creep was noted in first tests of up to 48-days duration or prior to rupture of the specimen; hence, it is assumed that logarithmic creep is the limiting type of long-duration response to stress.

The first-creep curves at different initial stresses could be combined to form a single generalized or master creep curve by reducing each curve in total deformation by factors proportional to the initial stress followed by shifts of the reduced curves along the log-time axis until they coincided in the regions of overlap. The required shifts in log time were stress-proportional. It was concluded, based on the master creep curve and the requirements for its construction, that the immediate elastic deformation is a linear function of initial stress and that one of the effects of increasing initial stress is a speeding up of the response. In order to form the master creep curve by this technique, it is necessary for the slopes of the logarithmic-creep portions of the first-creep curves to be linearly related to initial stress, and for the exponent of time in the exponential equations to be independent of initial stress.

It was noted that the rates of creep in creep and load-deformation tests were dependent solely on the existing total deformation and initial stress. At constant external conditions, creep rate may be independent of the path followed in arriving at a given point. It was felt that an apparent mechanical equation of state for continuous deformation in first tests may provide a useful basis for analyzing the load-deformation properties of paper at different rates of stress or strain development.

The amount of total creep recovery following first-creep tests increased with time of loading at a given initial stress until the onset

of logarithmic creep, after which no further increase in total recovery occurred despite further deformation in the logarithmic creep range. The transitional zone of creep is an area of rapidly decreasing recoverability. The limiting value of total first-recovery deformation was linearly related to initial stress; however, it was reached at shorter times at higher initial stresses in accordance with the time-shift requirement of the master creep curve concept. These results strengthen the observation that one of the effects of increasing initial stress is a speeding up of the response, and demonstrate that logarithmic creep deformation is nonrecoverable at the test conditions. It could not be demonstrated, however, that recoverable creep deformation would not occur in subsequent tests at times greater than the time at which logarithmic creep began in first-creep tests. This was one of several effects which indicated that creep in the logarithmic range had an inhibitory effect on recovery.

In second-creep tests at the same load, the total creep deformation at a time equal to the duration of the first test was lower than the total first-creep deformation--approximately by the amount of nonrecoverable deformation in the first test. Greater times of loading in first-creep tests caused progressive reductions in both creep rate and level of total deformation at early times in second-creep tests. As the second test was continued in time beyond the duration of the first test, the creep curve assumed the shape of the first-creep curve in the same range of time. The relationship between the early and long-duration response, therefore, is dependent on the previous mechanical history of the specimen. Thus, first-creep curves of widely different over-all shape can result

if the specimens are subjected to varying degrees of mechanical action before testing, or if similar changes in structure occur during drying of the wet sheets.

Three or more cycles of 24-hour-duration creep and recovery tests at the same initial stress are required before the response of subsequent tests becomes identical and the creep deformation is almost entirely recoverable upon removal of the load. A specimen in this condition is said to be mechanically conditioned. It was shown that part of the mechanical conditioning effect was an actual inhibition of previously recoverable deformation. This was shown by the fact that the creep recovery as well as the primary creep were reduced in both rate and level of total deformation in cyclic long-duration tests.

Primary-creep curves at different initial stresses appeared to be related in the same general manner as the first-creep curves, although in a different order of magnitude. For example, the nonlinearity of the total primary creep deformation versus initial stress relationship at various constant times was not as great. Primary creep curves exhibit exponential and logarithmic creep response in a time distribution analogous to first-creep curves. It appeared possible to construct master creep curves for primary creep, although the requirements for their construction could not be determined accurately because of the fact that the exact nature of the primary creep curve was a function of the severity of the mechanical conditioning tests.

At higher initial stresses, the recovery curve was displaced lower in total deformation than the preceding primary creep curve, but was

approximately parallel to it. At low initial stresses, the creep and recovery curves of long-duration tests were almost identical in shape and level of deformation over most of the experimental time interval. Thus, these handsheets may be said to obey Boltzmann's superposition principle at lower initial stresses, but not at higher stresses. It was felt, however, that agreement with Boltzmann's superposition principle was a matter of the extent and type of creep deformation, and that reasonable agreement with the principle would be obtained at all initial stresses for deformations before the onset of logarithmic and possibly transitional creep. At higher relative humidities, the same general behavior was noted, with the exception that the recovery curves were steeper in slope than the primary creep curves on the customary semilogarithmic plot in long-duration tests at higher initial stresses.

Greater time of loading in first-creep tests past the onset of the limiting value of recovery continues to affect the shape of the recovery curve. The total recovery deformation was reduced at earlier times, but the recovery curve became steeper at later times with increased time of loading. This behavior results from an inhibitory effect of logarithmic creep deformation on the recovery response by shifting the distribution of the recovery deformation toward longer times. Sufficient deformation in the logarithmic creep range may actually reduce the magnitude of the total recovery, particularly at the higher relative humidities.

The principal effects of handsheet solid fraction on creep response were changes in the initial stress required to reach specified total first-creep or total first-recovery deformations in given periods of time

by constant multiples between any two handsheets. At constant pulp freeness, an increase in solid fraction by wet pressing resulted in large decreases in creep response at lower solid fractions (at constant time and initial stress). The reduction in creep response was much lower at the higher solid fractions for similar increments of increasing solid fraction. The largest decreases in creep response with increasing solid fraction occur in a range where the smallest increases in optical bonded area will occur, whereas much larger increases in optical bonded area are expected in the range where creep response changes very slowly with solid fraction. This behavior was attributed to stress distribution effects which are more sensitive to the number of fiber-fiber contacts than to the total area of fiber-fiber bonding. In contrast, tensile strength increased almost linearly with increasing solid fraction. The effect of beating was principally a decrease in the creep response at constant solid fraction and initial stress. Slightly greater percentages of recovery were noted in long-duration tests for the handsheets prepared from the more highly beaten pulps. The first decreases in freeness of the pulp with beating appeared to be the most effective. Wet pressing had no effect on the percentage recovery of total first-creep deformations. The first-recovery curves were found to be identical in shape at equivalent levels of total first-recovery deformation, hence, the small differences in shape of the first-creep curves were attributable to the nonrecoverable component of the first-creep deformation.

Master creep curves could be constructed from the first-creep curves of handsheets of all solid fractions from 31.6 to 54.2% by the stress-

proportional technique. The time-shift requirements was lower for handsheets prepared from the more highly beaten pulps. As a first approximation, a change in sheet structure by beating or wet pressing could be represented as a change in an apparent fraction of the solid fibrous material capable of supporting the load.

Handsheets prepared from the on-20-mesh fraction of 775 cc. S.-R. freeness pulp were of approximately the same solid fraction and tensile strength as those prepared from the unfractionated pulp, but the response in creep and recovery tests was lower by about 20%. The handsheets showed reduced hygroexpansivity and less residual strain as indicated by the net shrinkages in an adsorption-desorption cycle. The percentages of recovery in first-creep and recovery tests were slightly lower. These data appear to be contradictory in attempting to attribute the effect of removal of fines to greater or lesser degrees of macroscopic tightness in sheet structure.

The principle effect of increasing relative humidity was a reduction in the initial stress required to reach specified levels of deformation in given periods of time. Up to about 75% R.H., this effect was due largely to a speeding up of all of the response. An abrupt decrease in the required stress occurred in the 50 to 63% R.H. range. This effect was also apparent as a rapid increase in the slope of the logarithmic-creep portion of the first-creep curves at constant initial stress in the same range of increasing relative humidity. The break in response versus relative humidity was attributed to a partial mechanical conditioning of the specimens at lower relative humidities as a result of drying under conditions of essentially complete planar restraint,

which was destroyed most rapidly at 55% R.H. by plasticization of the specimen. Similar breaks in response were not detected or either were very small in tests on the desorption curve following humidification of the specimens at 97% R.H.

At relative humidities below about 65 to 70%, it was possible to construct master creep curves with good fits in the regions of overlap by stress-proportional reductions of the curves in deformation plus shifts of the curves along the log-time axis. Greater time shifts were required per unit initial stress at higher relative humidities. The inability to construct the master creep curve from the first-creep curves at higher relative humidities was due to a reduced stress-dependency of the creep response plus a change in curve shape toward concave-downward response at the higher initial stresses just prior to logarithmic creep. The change in curve shape was more pronounced after humidification. This latter behavior suggests that the earlier response (preceding logarithmic creep) basically may follow a sigmoidal curve on the deformation versus log-time plot.

In tests on the desorption curve after humidification at 97% R.H., much greater total first-creep deformations were noted at 50 and 23.5% R.H. with practically no difference at 83% R.H. in comparison with the results on the adsorption curve before humidification. Further, the rates of creep at given long times in the logarithmic creep zone were the same between 50 and 83% R.H. at the same initial stress for the humidified specimens. It was expected that the identity in creep rate would apply over an even wider range of relative humidity. This effect was not noted in tests before humidification on the adsorption curve because of a change

in creep properties as a result of progressive recovery of the residual strain. It demonstrates an independence of the mechanisms of logarithmic creep on the moisture content of the specimen over rather wide ranges. Since the response preceding logarithmic creep increased in amount with increasing relative humidity, it was concluded that there are at least two basic types of response to stress in paper which are separated in time in constant-load creep tests and which vary in their relative-humidity dependence.

The percentage of recovery of the total or of the delayed first-creep deformation was at a minimum between 70 and 80% R.H. Part of the greater recoverability at the lower relative humidities could be traced to the partially mechanical-conditioned character of the specimens in that range of relative humidity; however, the increased recoverability at very high relative humidities could not be attributed to structural changes in the preparation of the specimens.

The nonrecoverable deformation in tests at 50% R.H. was recoverable in large measure by subjecting the specimens to a relative humidity of 97.8% followed by redrying to the test conditions. Practically all of the first-creep deformation to the onset of logarithmic creep and some of the logarithmic creep deformation was recoverable in this way. Continued logarithmic creep deformation, however, was resistant to recovery by humidification treatments.

A thorough interpretation of the creep and creep recovery behavior of these handsheets must necessarily be cautious, since the results of prerule mechanical tests are merely suggestive of the mechanisms of

response and their relation to the structure of the specimen. The response of a macroscopically heterogeneous material such as paper may include mechanisms of deformation which are not common to polymers of solid cross section. Many of these mechanisms may be of negligible importance in contributing to deformation and can be neglected. It is possible in this work to consider only those mechanisms of response which are rate controlling. One must assume that other effects do not influence the rate-controlling mechanisms in greater proportion than their possible contribution to the deformation of the specimen.

It is perhaps most significant that the general effect of initial stress on first-creep response indicates a degree of ideality that would not be expected if macroscopic uncurling or straightening of the fibers in the sheet were significant contributions to the total deformation. Similarly, it would require a most fortuitous combination of events to attribute the effect of changing solid fraction on creep behavior, for example, to mechanisms of this type. The first-creep properties of these handsheets are very similar to the primary creep properties if one accounts for the differences in degree of the effects. The entire pattern of creep and creep recovery behavior of these handsheets can be explained on the basis of rate-controlling molecular mechanisms of response and the differences in stress distribution throughout the solid fibrous material which are determined largely by the initial sheet structure. The following general interpretation is suggested. At relative humidities below about 70%, a tensile load applied to a specimen is distributed immediately in a nonuniform manner both between and within the load-transferring elements of fibrous structure. The early delayed response at any load

is due to the configurational rearrangement and increased alignment of the molecular chains in the amorphous areas of the polymer. A rather wide distribution of retardation times characterize this type of response and it is described at early times by an exponential equation. Greater degrees of intermolecular bonding, brought about by reducing the specimen moisture content, have the effect of increasing all of the retardation times with little effect on their distribution. Under constant external test conditions, the new configurational structure easily becomes fixed at new positions of metastable equilibrium either because of strong points of intermolecular bonding in the amorphous areas or because of steric hindrance effects, both of which have the effect of greatly increasing the retardation times for recovery. This type of response reaches a maximum which is linearly related to initial stress, but the response is also speeded up, which accounts for the nonlinearity of deformation-initial stress relationships at constant time and the apparent yield zone of stress. It is felt that this type of response follows a sigmoidal curve with an inflection point somewhere in the transitional zone of creep. When the retardation times are shifted sufficiently toward early times, the sigmoidal nature of this type of response in time becomes apparent. Near the onset of logarithmic creep, a straightening of the molecular chains has occurred to the degree that further response to stress is contingent on the necessity for rotation of the molecular chains in the fringe areas. This will account for small increases in the degree of crystallinity of the polymer. The rate of this longer-duration response may be controlled at the lower relative humidities by the continuation of configurational response of long retardation times, but it would appear

that without additional crystallization, this type of response would not occur. At higher relative humidities, the retardation times are shortened sufficiently for the configurational response to occur very rapidly in time. After the essential completion of the possible configurational elastic response, crystallization occurs, but the rate becomes less dependent on initial stress because of a reduced necessity for configurational response. The driving force for crystallite growth apparently does not increase with stress at high relative humidities.

Plasticization of the specimen by increasing the moisture content reduces the intermolecular bonding and separates the molecular chains to permit practically complete recovery of the configurational elastic deformation plus a part of the stress-induced crystallization. The humidification of the as-dried specimens provides for similar effects in the recovery of residual strains resulting from drying under tension; however, these structural changes occur over ranges of relative humidity and are complex.

These data indicate that part of the difference in response between handsheets of different solid fraction may be due to changes in molecular structure during drying which differ in magnitude for the different handsheets and can be related chiefly to the degree of beating. It is postulated, however, that the chief effect of changing solid fraction is a change in stress distribution throughout the sheet, which either changes very little with deformation or which changes in the same manner for the different handsheets. The exact nature of the stress distribution effect cannot be suggested. It is a large effect but has a negligible effect on the pattern of creep and recovery. After recovery of residual strain, the effect may be even smaller.

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APPENDIX

TABLE A

TIME-DEFORMATION DATA IN CREEP AND RECOVERY TESTS

All of the time versus deformation data pertinent to this study are presented in this table in order of test number. The time is given in seconds measured from the instant of application or removal of load. The deformations in creep or recovery tests are total values measured from the specimen length just prior to the application or removal of load. The deformations are reported in mils (inches x 1000). In all tests, the initial specimen dimensions were 10.00 inches in length and 1.00 inch in width at 50% R.H. The deformation in percentage of the initial length may be calculated easily as mils/100. The following test identification is employed.

Specimen number, as indicated
Load, total load in creep test in grams
Stress, calculated initial stress in kg./sq. mm.
Recovery load, no load unless residual load is indicated
R.H., relative humidity in per cent

The following abbreviations were employed.

a.d.,	as-dried
ads.,	adsorption
des.,	desorption
300 g.d.,	300-gram deformation, which represents the additive correction factor due to the weight of the lower clamp assembly in obtaining readings with the creep testing units.

All tests were run at a nominal temperature of 73°F.

TABLE A

TIME-DEFORMATION DATA IN CREEP AND RECOVERY TESTS

TEST 11				TEST 12	
Specimen 21-7		5530	46.2	Specimen 21-6	
Load, 3500		6370	46.7	Load, 3500	
Stress, 3.64		44,290	51.9	Stress, 3.64	
R.H., 50% a.d.		54,850	52.7	R.H., 50% a.d.	
300 g.d., 3.0 mils		85,200	53.1	300 g.d., 3.0 mils	
First-creep		138,600	54.1	First-creep	
Seconds	Mils	247,200	55.3	Seconds	Mils
		392,600	56.2		
15	44.0	606,300	56.8	17	45.2
40	47.4	701,200	57.5	55	49.1
64	49.0	963,600	57.7	81	50.6
87	50.2	1,437,000	58.1	135	52.8
139	52.2	1,774,000	57.8	230	55.4
173	53.2	Second-creep		368	57.8
360	57.1	Total stress, 3.64		624	60.3
435	58.0	Added stress, 3.33		1065	64.1
656	59.9	Seconds	Mils	1555	66.1
980	62.4			2020	68.4
1410	64.9	10	33.0	3585	72.4
3370	71.4	31	34.3	4785	75.1
6780	76.9	51	35.0	7125	78.3
11,100	81.5	102	36.0	20,200	88.3
13,380	83.5	197	37.0	21,830	89.1
26,340	90.3	382	38.2	29,800	92.1
29,880	91.6	1100	40.3	68,760	100.9
30,000	Recovery	2205	42.1	109,300	105.3
First-recovery		7500	45.7	162,300	109.1
Residual load, 300 grams		18,720	49.1	271,100	114.6
		39,360	52.6	416,500	118.7
Seconds	Mils	86,820	57.4	526,700	121.1
		137,500	60.0	544,800	121.3
14	34.0	176,900	61.9	587,000	122.1
45	35.2	283,400	65.2	587,100	Recovery
76	36.1	387,600	68.2		
111	36.5	468,700	70.0		
157	38.1	625,300	72.6		
216	38.8	697,300	73.5		
350	39.7				
540	40.4				
776	41.4				
1210	42.5				

TABLE A (continued)

TIME-DEFORMATION DATA IN CREEP AND RECOVERY TESTS

TEST 12		155,600	50.0	324	42.6
(continued)		216,500	51.2	550	43.3
		311,800	52.9	995	43.4
Residual load,		341,000	53.1	1700	45.0
300 grams		497,400	54.6	14,520	47.1
		657,500	55.6	19,260	47.4
Seconds	Mils	697,800	56.3	24,480	47.6
				63,000	47.9
11	30.7	TEST 13		73,140	48.4
31	31.6			103,200	48.5
61	32.3	Specimen 21-2		156,700	48.6
144	33.3	Load, 3500		265,300	48.9
412	35.0	Stress, 3.64		410,700	49.3
900	36.1	R.H., 50% a.d.		521,000	49.4
1395	37.4	300 g.d., 3.0 mils		667,400	49.6
1945	37.9			762,500	49.7
8760	41.0	First-creeep		927,700	49.8
16,140	42.9			1,025,000	49.9
23,460	43.4	Seconds	Mils	1,455,000	50.1
85,740	47.2	36	46.8		
181,100	49.7	66	48.8	Second-creeep	
346,100	51.5	92	50.0		
444,200	52.6	163	52.1	Total stress, 3.64	
519,800	53.1	253	53.9	Added stress, 3.32	
614,800	54.3	367	55.6		
719,200	54.6	492	57.0	Seconds	Mils
1,040,000	54.5	687	58.7		
1,251,000	54.9	886	60.5	37	35.3
		1048	61.6	68	36.3
Second-creeep		1555	63.9	124	37.3
		1742	65.2	278	39.0
Total stress, 3.64		1885	65.5	535	40.1
Added stress, 3.33		1995	65.8	1182	42.3
		2000	Recovery	5280	48.5
Seconds	Mils			13,800	53.2
		First-recovery		23,700	56.6
23	31.9			47,160	61.5
53	32.8	Residual load,		84,120	66.4
86	33.2	300 grams		109,000	70.0
157	33.9			170,300	74.3
376	35.2	Seconds	Mils	210,400	76.3
770	36.2			257,600	78.4
2557	38.2	26	38.1	347,300	81.3
9420	41.0	80	40.0	454,500	83.9
48,480	45.7	113	40.8	558,300	86.3
		231	42.1	639,800	87.4
				697,100	88.5

TABLE A (Continued)

TIME-DEFORMATION DATA IN CREEP AND RECOVERY TESTS

TEST 14

Specimen 21-3	592,300	43.0
Load, 3500	852,500	43.4
Stress, 3.64	1,026,000	43.4
R.H., 50% a.d.	1,485,000	43.4
300 g.d., 3.0 mils	1,640,000	42.9

First-creep

Seconds Mils

15	43.3
33	46.0
52	47.4
72	48.4
93	49.1
123	50.0
152	50.7
176	51.1
200	Recovery

First-recovery

Residual load,
300 grams

Seconds Mils

14	38.1
43	39.5
65	40.0
87	40.4
141	40.8
228	41.0
492	41.4
920	41.8
2920	42.3
7300	42.7
14,500	42.9
27,700	42.7
81,520	42.6
190,200	42.8
335,600	42.9
445,700	43.1
465,100	43.0

Second-creep

Total stress, 3.64
Added stress, 3.33

Seconds Mils

22	36.3
48	38.0
85	39.1
149	40.1
262	41.3
382	42.3
557	43.3
2437	49.6
15,180	62.3
76,860	74.5
116,900	79.5
164,000	83.2
253,900	87.9
361,000	91.2
464,600	93.8
546,400	95.3
697,000	97.9

TABLE A (Continued)

TIME-DEFORMATION DATA IN CREEP AND RECOVERY TESTS

TEST 15		TEST 16		TEST 17	
Specimen 23-7		Specimen 23-8		Specimen 23-9	
Load, 4500		Load, 4500		Load, 3500	
Stress, 4.63		Stress, 4.62		Stress, 3.60	
R. H., 50% a.d.		R.H., 50% a.d.		R.H., 50% a.d.	
300 g.d., 3 mils		300 g.d., 3 mils		300 g.d., 3 mils	
First-Creep		First-Creep		First-Creep	
Seconds	Mils	Seconds	Mils	Seconds	Mils
45	89.5	11	77.8	15	46.6
161	101.8	32	86.3	40	49.2
193	103.5	57	91.5	72	51.1
260	106.5	115	98.2	126	53.2
396	111.4	161	101.8	177	54.4
487	113.8	206	104.5	233	56.0
670	117.6	248	106.5	402	58.9
930	121.4	389	112.0	948	63.3
1440	127.0	545	116.0	1420	65.7
1920	130.7	710	119.0	2490	69.4
3000	136.8	950	122.3	3810	72.1
4440	141.8	1770	130.0	6150	75.6
7500	148.6	3420	138.5	15,270	84.1
9780	152.4	6240	145.9	27,750	89.7
18,480	161.8	8520	150.5	66,330	98.6
30,840	169.1	17,460	161.0	92,130	102.4
69,300	181.5	29,880	168.4	152,600	108.6
95,400	186.8	68,340	181.1	202,700	111.2
155,600	193.8	94,320	186.7	245,600	113.2
205,900	197.0	154,700	193.6	332,400	116.3
248,800	199.7	204,800	196.9	421,200	119.0
335,400	203.5	247,700	199.8	522,600	121.4
424,300	206.5	334,400	203.5	626,000	122.9
525,800	209.9	423,300	206.7	758,100	124.7
629,000	212.3	524,700	210.3	864,000	126.1
761,400	214.7	628,100	212.3		
863,900	216.5	760,300	215.0		
		864,000	216.6		

TABLE A (Continued)

TIME-DEFORMATION DATA IN CREEP AND RECOVERY TESTS

TEST 18		TEST 19		TEST 20	
Specimen 23-10		Specimen 23-13		Specimen 23-6	
Load, 3500		Load, 4500		Load, 2000	
Stress, 3.60		Stress, 4.68		Stress, 2.06	
R.H., 50% a.d.		R.H., 50% a.d.		R.H., 50% a.d.	
300 g.d., 3 mils		300 g.d., 3 mils		300 g.d., 3 mils	
First-creeep		First-creeep		First-creeep	
Seconds	Mils	Seconds	Mils	Seconds	Mils
11	44.7	9	76.3	16	21.3
38	48.1	34	86.8	39	21.8
96	51.2	68	93.6	142	22.8
138	52.6	118	99.1	275	23.4
210	54.0	193	104.6	647	23.7
380	56.8	239	107.1	960	24.4
745	60.2	294	109.6	1640	25.0
915	61.3	391	112.9	3840	26.3
1240	63.2	491	115.3	13,560	28.4
1686	65.1	879	121.8	26,340	30.2
3070	68.5	1390	126.9	65,220	32.7
5400	72.6	2385	133.8	90,360	34.6
14,700	81.0	4965	143.1	151,000	36.2
27,420	87.0	7065	147.7	201,100	36.7
66,180	95.6	16,545	159.5	243,900	37.6
91,500	99.6	29,265	167.9	330,900	38.9
152,000	105.1	68,085	179.8	419,600	40.1
202,100	107.4	93,290	185.1	520,900	41.5
245,000	109.6	153,800	192.7	624,700	42.2
331,900	113.0	203,900	195.8	756,500	43.4
420,700	115.3	246,800	198.0	864,000	43.7
522,100	117.9	333,800	201.6		
625,600	119.3	422,500	204.7		
757,600	121.2	523,800	208.0		
864,000	122.8	627,500	210.4		
		759,400	212.8		
		864,000	214.5		

TABLE A (Continued)

TIME-DEFORMATION DATA IN CREEP AND RECOVERY TESTS

TEST 21		Second-creep Stress, 3.64		TEST 22	
Specimen 24-9				Specimen 24-8	
Load, 3500				Load, 3500	
Stress, 3.64				Stress, 3.61	
R.H., 50% a.d.				R.H., 50% a.d.	
300 g.d., 3.0 mils				300 g.d., 3.0 mils	
First-creep				First-creep	
Seconds	Mils	Seconds	Mils	Seconds	Mils
13	42.7	14	37.9	25	42.6
32	44.7	58	39.9	48	44.0
51	45.7	435	43.8	71	45.0
80	46.9	655	44.8	99	45.9
261	50.8	1114	45.3	163	47.3
590	53.9	1742	46.4	325	50.0
951	55.9	2044	46.7	631	52.5
1860	59.0	4410	48.7	1125	55.1
9120	68.9	9570	51.2	8370	66.3
21,100	75.3	23,200	54.3	20,400	72.3
65,900	85.3	76,300	59.5	65,200	82.5
144,900	92.9	116,000	61.3	144,200	90.1
173,600	94.5	276,900	66.9	172,900	91.8
182,800	95.2	370,600	69.2	182,900	92.5
183,000	Recovery	543,600	72.4	183,000	Recovery
		592,700	73.5		
		592,900	Recovery		
		Second-recovery			
		Residual load,			
		300 grams			
First-recovery				First-recovery	
Seconds	Mils	Seconds	Mils	Seconds	Mils
106	39.1	12	31.4	15	32.4
430	41.6	38	32.5	36	33.4
1200	44.6	173	34.4	61	33.9
2580	46.1	300	34.9	107	34.6
4500	47.6	435	35.6	154	35.0
9960	49.9	738	36.6	276	35.9
57,200	54.8	1250	37.4	418	36.7
89,500	56.2	4260	39.6	945	38.2
135,800	57.3	16,800	42.4	1695	39.5
220,100	58.7	35,600	44.6	2580	40.6
259,900	58.7	82,400	46.5	3420	41.1
299,300	59.0	121,200	47.3	9240	43.5
351,600	59.4	191,700	48.6	56,400	47.7
401,900	60.1	345,800	49.8	88,700	48.9
568,700	61.0	515,600	51.0	135,100	49.9
756,600	61.3	786,100	51.6	219,400	50.7
999,600	61.3	947,100	51.8	298,600	51.5
1,165,000	62.2	1,000,000	52.1	401,200	52.2
1,431,000	62.4				
1,447,000	Reload				

TABLE A (Continued)

TIME-DEFORMATION DATA IN CREEP AND RECOVERY TESTS

TEST 22 (continued)		TEST 23		Second-creep	
				Total stress, 3.60	
				Added stress, 2.98	
478,700	52.5	Specimen 24-7			
669,000	53.0	Load, 3500			
755,900	53.2	Stress, 3.60			
998,900	53.7	R.H., 50% a.d.			
1,164,000	53.8	300 g.d., 3.0 mils			
1,448,000	54.5				
Second-creep		First-creep			
Total stress, 3.61					
Added stress, 3.30					
Seconds	Mils	Seconds	Mils	Seconds	Mils
23	33.2	37	43.5	12	28.0
67	34.6	68	45.2	38	28.9
97	35.0	101	46.3	119	29.9
166	35.8	136	47.4	290	30.9
270	36.3	193	48.7	510	31.9
777	37.9	511	52.1	970	32.9
1905	39.6	7590	66.1	1715	33.7
3165	40.9	19,800	72.8	2640	34.3
8085	43.2	64,600	82.9	5400	36.0
21,900	46.1	143,600	90.6	9960	37.1
74,900	51.0	172,200	92.2	24,200	39.9
114,700	52.5	182,900	92.8	76,900	43.9
275,600	57.7	183,000	Recovery	116,900	45.1
369,500	60.0	First-recovery		277,800	51.0
542,400	63.9	Residual load,		371,800	53.1
589,100	64.3	600 grams		544,600	56.9
592,100	Recovery			591,400	57.7
Second-recovery				595,000	Recovery
				Second-recovery	
				Residual load,	
				600 grams	
Seconds	Mils	Seconds	Mils	Seconds	Mils
120	38.0	30	28.9	16	27.7
510	40.5	59	29.6	47	28.6
1125	41.8	92	29.9	73	28.9
3480	43.9	160	30.6	146	29.7
16,100	47.8	401	31.8	300	30.2
35,000	50.0	923	33.0	840	31.2
81,400	52.0	2890	35.3	2620	33.0
120,500	53.6	8770	37.5	15,300	35.7
191,300	54.7	55,600	41.0	34,400	37.5
345,400	56.2	88,200	41.9	80,700	38.7
515,200	57.5	134,700	42.9	119,900	39.8
780,900	58.5	218,900	43.7	190,600	40.7
946,200	58.8	258,800	43.9	344,800	41.6
		298,000	44.2	514,600	42.4
		400,600	45.1	783,700	42.7
		478,100	45.4	945,600	42.9
		567,400	45.5	1,032,000	43.3
		668,400	45.7	1,037,000	43.6
		755,400	45.9		
		998,400	46.5		
		1,164,000	46.5		
		1,445,000	46.6		

TABLE A (Continued)

TIME-DEFORMATION DATA IN CREEP AND RECOVERY TESTS

TEST 24		TEST 25		940	252.8
Specimen 23-2		Specimen 23-4		1271	259.1
Load, 5500		Load, 6000		1452	261.8
Stress, 5.69		Stress, 6.20		1695	264.6
R.H., 50% a.d.		R.H., 50% a.d.		1770	265.5
300 g.d., 3 mils		300 g.d., 3 mils		2188	269.5
First-creeep		First-creeep		2782	273.5
Seconds	Mils	Seconds	Mils	3315	276.5
14	121.3	20	179.0	4156	280.5
35	134.0	35	190.5	5365	285.5
49	139.1	51	197.1	6565	289.5
63	142.8	70	203.0	10,470	298.5
81	146.3	88	207.6	12,450	301.8
100	149.2	111	211.0	15,930	rupture
171	157.0	134	215.2	TEST 27	
199	159.4	155	217.8	Specimen 23-1	
254	162.8	257	226.4	Load, 5500	
304	165.6	290	228.4	Stress, 5.70	
358	167.8	345	231.3	R.H., 50% a.d.	
443	170.8	454	236.5	300 g.d., 3 mils	
548	173.8	683	243.9	First-creeep	
840	180.8	890	249.2	Seconds Mils	
1205	185.8	1134	253.6	15	127.8
1635	191.0	1388	257.1	32	140.1
2217	195.6	1671	261.1	46	145.4
2812	198.8	2152	265.5	60	149.3
4300	205.6	2510	rupture	76	153.0
5385	209.2	TEST 26		107	158.3
7305	214.1	Specimen 23-11		162	165.3
9345	218.1	Load, 6000		235	171.2
10,430	219.8	Stress, 6.19		317	176.3
12,650	222.8	R.H., 50% a.d.		363	178.6
14,450	224.8	300 g.d., 3 mils		420	181.3
21,890	231.4	First-creeep		465	183.2
27,650	235.3	Seconds Mils		620	190.3
31,070	237.0	20	178.4	767	192.3
34,070	238.7	43	193.5	948	195.3
73,790	250.6	63	200.5	1187	199.3
95,990	255.3	93	208.4	1495	203.3
117,300	258.3	216	224.2	2085	209.0
136,900	rupture	520	241.7	2900	215.2
				4920	223.6
				10,260	236.0
				17,940	245.9
				34,080	256.3
				- - -	rupture

TABLE A (Continued)

TIME-DEFORMATION DATA IN CREEP AND RECOVERY TESTS

TEST 29	3,366,000	227.3
	3,485,000	227.3
Specimen 42-1	3,658,000	228.4
Load, 4500	3,879,000	229.4
Stress, 4.90	3,985,000	230.3
R.H., 50% a.d.	4,164,000	rupture
300 g.d., 3.0 mils		

First-creep

Seconds	Mils
21	73.9
46	79.3
77	83.2
166	89.4
270	93.5
462	98.8
1417	112.1
3385	121.5
11,700	138.3
33,600	153.9
74,220	165.9
114,000	172.5
162,300	178.3
246,300	184.6
440,000	192.8
510,900	198.0
695,900	200.5
764,600	202.3
1,028,000	206.5
1,245,000	209.9
1,370,000	211.0
1,455,000	212.0
1,670,000	213.7
1,803,000	215.1
1,891,000	216.1
2,021,000	217.2
2,105,000	218.3
2,274,000	220.8
2,433,000	221.4
2,602,000	222.8
2,799,000	223.4
2,962,000	224.2
3,201,000	226.3

TABLE A (Continued)

TIME-DEFORMATION DATA IN CREEP AND RECOVERY TESTS

TEST 34		1577	46.0	First-recovery	
		3215	47.9		
Specimen 28-4		13,320	52.0	Seconds	Mils
Load, 3500		64,440	56.8		
Stress, 3.62		155,000	57.7	95	43.7
R.H., 50% a.d.		239,900	60.7	200	45.2
300 g.d., 3 mils		343,400	62.2	421	46.4
		433,100	63.3	815	48.6
First-creep		627,600	64.1	992	49.1
		798,500	65.2	1460	50.0
Seconds	Mils	864,000	65.3	2575	51.9
11	47.3	TEST 35		11,160	56.8
38	51.3			14,580	58.0
61	53.3	Specimen 29-3		19,740	58.8
85	54.8	Load, 3500		58,980	62.2
124	56.2	Stress, 3.74		89,520	63.1
198	58.1	R.H., 50% a.d.		143,300	64.7
244	59.0	300 g.d., 3 mils		185,000	65.9
477	61.8	First-creep		259,100	66.5
925	65.5			TEST 36	
1694	69.2			Specimen 30-11	
2415	71.3	Seconds		Load, 3500	
3990	75.2	Mils		Stress, 3.65	
11,100	83.3	19	51.0	R.H., 50% a.d.	
16,660	87.1	41	53.8	300 g.d., 3 mils	
21,100	89.4	56	55.4	First-creep	
58,380	100.0	115	58.0		
92,860	105.3	156	59.4		
139,400	109.7	250	61.7	Seconds	
187,000	112.6	428	64.3	Mils	
233,600	115.1	810	67.5		
318,900	118.4	1628	72.0	18	47.4
447,000	122.1	3330	77.2	42	49.8
533,300	124.2	10,320	86.5	90	52.7
581,800	125.1	15,840	90.3	124	53.9
756,000	129.2	20,280	92.8	175	55.6
864,100	131.5	57,600	103.8	322	58.3
		97,080	109.0	388	59.2
First-recovery		147,000	113.5	560	61.3
		194,600	116.5	1029	64.6
Seconds	Mils	232,900	118.9	2850	71.1
		258,800	120.1	9720	80.7
101	39.6			15,240	84.6
250	41.3			19,680	86.9
760	43.6			57,000	97.5

TABLE A (Continued)

TIME-DEFORMATION DATA IN CREEP AND RECOVERY TESTS

TEST 36 (continued)		TEST 37		TEST 38	
First-creep		Specimen 31-9 Load, 3500 Stress, 3.61 R.H., 50% a.d. 300 g.d., 3 mils		Specimen 32-8 Load, 3500 Stress, 3.69 R.H., 50% a.d. 300 g.d., 3 mils	
Seconds	Mils	First-creep		First-creep	
100,100	102.6	Seconds	Mils	Seconds	Mils
146,600	107.0	11	45.7	16	47.5
194,200	109.8	36	49.3	35	49.7
232,200	112.0	57	50.5	50	50.8
317,700	115.3	95	52.3	69	51.8
360,700	117.9	152	54.0	109	53.4
402,500	118.5	371	58.3	173	54.9
445,500	120.7	820	62.3	266	56.6
494,200	121.4	1599	66.1	435	59.0
531,700	122.4	2160	68.1	570	60.9
753,600	125.5	2785	69.4	934	63.0
864,000	128.2	4770	73.2	1499	65.8
First-recovery		9240	78.2	2130	67.8
Seconds	Mils	12,840	81.0	4068	72.1
102	39.6	46,560	93.3	8580	77.8
249	41.5	86,340	98.8	12,180	80.8
796	43.8	First-recovery		45,840	93.1
1830	46.0	Seconds	Mils	85,380	98.8
11,880	51.2	97	43.9	135,500	103.8
63,120	57.4	287	46.5	193,200	106.9
153,600	59.9	657	48.6	221,200	109.1
238,700	61.8	2000	51.3	259,200	110.5
342,000	62.8	4930	53.6	First-recovery	
431,800	63.8	10,810	56.2	99	41.7
593,600	64.9	49,440	60.5	244	43.6
797,300	66.0	75,180	61.3	438	44.7
963,500	66.5	86,340	61.9	667	45.9
1,484,000	67.5			1087	47.0
1,530,000	67.4			1883	48.6
				2871	49.4
				7735	52.8
				47,760	58.4
				78,180	59.6
				133,600	61.2
				175,400	62.5
				259,100	63.4

TABLE A (Continued)

TIME-DEFORMATION DATA IN CREEP AND RECOVERY TESTS

TEST 39		Second-creep		356	47.9
				1474	51.0
Specimen 33-8		Stress, 3.64		3420	52.8
Load, 3500				45,720	60.4
Stress, 3.64		Seconds	Mils	86,340	62.8
R.H., 50% a.d.				Third-recovery	
300 g.d., 3 mils		26	43.5		
		50	44.8		
First-creep		101	46.3	Seconds	Mils
		240	48.7		
Seconds	Mils	587	50.8	101	41.7
6	45.0	978	52.0	228	43.3
32	49.0	1520	53.3	1162	47.4
83	52.0	3610	56.0	73,440	57.6
122	53.3	9000	59.0	86,460	57.7
150	54.2	46,800	65.0	Fourth-creep	
188	55.2	72,120	67.9	Stress, 3.64	
356	58.3	85,020	68.2		
814	62.5	86,340	68.5		
1006	63.8	Second-recovery		Seconds	Mils
2702	70.5				
7200	76.5	Seconds	Mils	25	42.0
10,800	80.8			52	43.3
44,460	94.5	98	42.0	73	43.8
86,340	100.7	208	44.5	110	44.3
First-recovery		382	46.0	173	45.1
		602	46.8	292	46.1
Seconds	Mils	1465	48.7	452	47.1
115	44.1	6325	52.7	850	48.5
272	46.2	46,560	58.2	917	48.3
700	48.9	76,920	59.1	1391	49.5
2980	52.7	86,340	58.9	2005	50.1
10,100	56.0	Third-creep		49,980	58.0
47,640	60.5	Stress, 3.64		86,340	60.0
73,080	61.7			Fourth-recovery	
86,340	62.5	Seconds	Mils	Seconds	Mils
		18	41.5	99	41.7
		37	42.9	355	43.9
		55	43.5	731	44.9
		105	45.1	2615	48.3
		197	46.3	6220	50.3
				46,980	55.3
				86,340	56.6

TABLE A (Continued)

TIME-DEFORMATION DATA IN CREEP AND RECOVERY TESTS

TEST 39 (continued)		320	46.4	Seventh-recovery	
		546	47.4	Seconds	Mils
		818	48.1		
Fifth-creep		1043	48.6		
		2835	50.4	101	42.2
Stress, 3.64		7500	52.9	255	44.2
		50,820	58.2	1115	46.4
Seconds	Mils	75,060	59.1	3065	48.6
		86,340	59.5	7800	50.7
11	40.5			62,760	55.1
31	41.9	Sixth-recovery		95,580	56.3
60	42.8	Seconds	Mils	266,900	57.9
117	43.9			433,000	58.5
211	44.8			953,100	60.3
350	46.1	120	42.3	1,453,100	60.0
2200	49.0	328	44.6	2,467,200	62.7
4680	51.7	1070	46.9		
49,740	57.8	2740	49.1		
68,460	59.6	4760	50.2		
86,340	60.0	54,840	55.4		
		140,300	57.3		
Fifth-recovery		174,300	57.9		
Seconds	Mils	243,200	58.6		
		259,100	58.8		
103	42.3	332,800	59.3		
275	44.1	Seventh-creep			
727	45.7	Stress, 3.64			
1735	47.6	Seconds	Mils		
2809	48.3				
9300	51.3	12	40.2		
49,020	55.4	35	41.6		
86,340	56.9	54	42.2		
Sixth-creep		113	43.4		
		188	44.4		
Stress, 3.64		381	45.7		
Seconds	Mils	960	47.5		
		3480	50.6		
23	42.0	8940	53.2		
45	42.9	11,160	54.1		
84	43.7	46,680	57.7		
170	45.2	86,280	59.5		
259	45.9				

TABLE A (Continued)

TIME-DEFORMATION DATA IN CREEP AND RECOVERY TESTS

TEST 40		First-recovery		Second-recovery	
Specimen 32-3		Seconds	Mils	Seconds	Mils
Load, 3500					
Stress, 3.65		108	65.9	105	65.2
R.H., 83%		258	71.2	258	69.0
Reached by ads.		492	74.7	546	72.4
from 12% R.H.		1505	80.7	1640	78.0
300 g.d., 4.5 mils		2305	83.2	4420	82.6
		3864	85.8	13,020	87.3
First-creep		8220	89.9	40,920	92.3
		18,360	95.4	86,340	95.5
Seconds	Mils	23,760	96.4	Third-creep	
13	152.0	42,360	99.5		
38	170.6	80,220	102.7		
60	178.6	86,400	103.5		
79	183.9	Second-creep		Stress, 3.65	
100	188.9	Stress, 3.65		Seconds	Mils
125	193.3			11	66.0
149	196.5			27	69.8
177	199.7	Seconds	Mils	48	72.3
202	202.1			85	74.7
328	210.1	15	69.4	123	76.5
452	216.6	38	73.5	236	78.7
527	218.5	61	75.6	585	82.1
575	219.7	113	78.5	1970	85.8
771	224.2	171	80.4	2740	87.2
1157	231.8	262	82.1	4470	88.7
1520	235.8	394	83.5	5940	89.5
2100	240.1	606	85.5	14,100	92.6
2668	243.6	980	87.7	34,680	95.2
3337	249.2	1480	89.2	81,420	99.5
4945	252.5	2136	90.3	86,340	99.6
7685	258.8	3642	93.2	Third-recovery	
9690	264.2	8880	97.2		
15,810	274.3	18,060	100.8		
18,690	276.6	32,940	105.4		
19,350	277.4	42,900	107.8	Seconds	Mils
24,210	280.3	84,180	112.2	109	63.6
35,130	286.1	86,340	112.3	251	67.8
42,030	289.6			500	70.7
79,650	301.2			1285	74.8
86,400	301.7			1923	76.9
				3230	78.9

TABLE A (Continued)

TIME-DEFORMATION DATA IN CREEP AND RECOVERY TESTS

TEST 40 (continued)		Fifth-creep		Sixth-creep	
Third-recovery		Load, 2800 Stress, 2.92		Load, 2000 Stress, 2.08	
Seconds	Mils	Seconds	Mils	Seconds	Mils
6000	81.6	15	47.8	14	30.9
12,480	84.9	36	50.6	26	31.9
22,860	87.1	59	51.8	51	33.3
33,720	88.6	84	52.9	69	33.8
80,940	91.6	180	55.4	123	35.1
86,400	91.8	406	57.0	257	36.6
		750	59.5	1815	40.9
Fourth-creep		1527	60.7	2315	41.5
		2250	62.4	5970	42.6
Stress, 3.65		3160	63.4	7005	43.7
		4085	64.0	16,260	45.7
Seconds	Mils	9000	66.5	85,620	49.4
		18,340	66.9		
14	65.7	23,440	68.3	Sixth-recovery	
44	70.0	42,520	69.0	Seconds	Mils
80	72.0	83,620	71.0		
161	74.5	86,340	70.9	97	32.2
367	77.3			260	34.2
960	80.6	Fifth-recovery		915	37.2
1888	82.6	Seconds	Mils	2300	39.5
3660	84.1			35,700	46.0
10,500	87.2	112	46.9	83,460	48.2
17,820	88.3	255	49.9	86,280	48.3
33,900	91.5	820	54.3		
82,320	94.8	1710	56.5	Seventh-creep	
86,400	95.1	2745	58.2		
		8380	61.8	Load, 1300	
Fourth-recovery		16,680	63.6	Stress, 1.35	
Seconds	Mils	24,900	65.2		
		38,040	67.2	Seconds	Mils
96	63.2	47,400	67.4		
530	70.5	86,280	69.5	22	18.4
965	73.1			45	18.7
2645	78.0			89	19.4
4815	79.1			208	20.2
10,980	82.3			390	20.5
35,880	86.6			3540	23.7
83,220	89.0			8940	25.2
86,340	89.5			24,000	26.6

TABLE A (Continued)

TIME-DEFORMATION DATA IN CREEP AND RECOVERY TESTS

TEST 40 (continued)		3280	268.2	176	144.8
		5635	275.1	228	148.4
		7980	272.3	302	152.6
Seventh-creep		13,980	293.8	368	156.0
		16,860	296.5	530	161.5
Seconds	Mils	22,380	300.5	919	169.2
		33,300	307.3	1511	180.5
81,180	28.3	40,200	307.0	1700	183.2
86,340	28.5	77,820	313.2	1751	183.8
		86,400	314.3	1819	184.7
Seventh-recovery		First-recovery		1881	185.4
				1935	186.0
Seconds	Mils			1984	186.3
		Seconds	Mils	2025	186.9
97	18.7			3802	198.6
350	20.2	105	69.6	6180	206.1
840	21.1	296	75.3	17,040	222.5
4620	23.4	505	78.1	24,000	229.0
22,320	26.0	1015	82.0	61,560	245.1
34,200	26.6	2180	86.2	72,600	246.5
86,700	28.0	6420	91.8	86,340	248.9
		16,560	97.3	First-recovery	
		21,960	99.2		
TEST 41		40,680	102.4	Seconds	Mils
Specimen 30-85		78,780	106.0		
Load, 3500		86,220	107.2	103	57.8
Stress, 3.68		TEST 42		488	64.9
R.H., 83%		Specimen 31-7		625	67.6
Reached by ads.		Load, 2800		966	69.8
from 12% R.H.		Stress, 2.91		2020	73.9
300 g.d., 4.5 mils		R.H., 83%		5760	79.9
First-creep		Reached by ads.		24,660	86.3
		from 12% R.H.		62,640	91.0
Seconds	Mils	300 g.d., 4.5 mils		77,520	92.5
		First-creep		86,340	92.7
45	196.6				
74	205.5				
122	215.0				
173	221.5				
208	225.0				
254	228.5				
354	234.6				
537	241.8				
783	248.3				
1405	257.5				
2128	263.2				

TABLE A (Continued)

TIME-DEFORMATION DATA IN CREEP AND RECOVERY TESTS

TEST 43		TEST 44		TEST 45	
Specimen 32-2		Specimen 28-2		Specimen 33-6	
Load, 2000		Load, 2000		Load, 3500	
Stress, 2.09		Stress, 2.07		Stress, 3.62	
R.H., 83%		R.H., 83%		R.H., 83%	
Reached by ads.		Reached by ads.		Reached by ads.	
from 12% R.H.		from 12% R.H.		from 12% R.H.	
300 g.d., 4.5 mils		300 g.d., 4.5 mils		300 g.d., 4.5 mils	
First-creep		First-creep		First-creep	
Seconds	Mils	Seconds	Mils	Seconds	Mils
12	45.8	18	51.5	17	222.8
25	49.2	51	57.7	35	237.6
38	51.7	72	60.5	57	247.5
58	53.8	135	65.4	73	252.4
128	59.3	220	69.2	92	257.2
194	62.6	316	72.3	110	260.9
258	64.6	606	78.0	128	264.8
588	71.1	890	81.7	150	268.7
973	75.6	1207	85.3	247	279.5
2180	83.4	2248	91.5	308	284.3
4108	89.5	4928	101.4	505	295.6
8640	97.8	8100	106.3	840	305.4
24,420	111.7	11,220	113.3	1313	314.2
43,080	120.6	13,680	115.5	1752	319.2
80,880	127.8	24,240	124.4	2820	326.2
86,460	128.0	31,200	128.5	4200	331.5
		68,760	140.8	5280	334.8
		80,040	142.0	6780	338.3
		86,460	143.1	7740	340.5
First-recovery		First-recovery		First-recovery	
Seconds	Mils	Seconds	Mils	Seconds	Mils
103	37.9			10,620	345.8
185	40.2			15,000	351.0
632	42.0			22,500	357.1
1265	46.9	96	40.2	45,180	369.0
2520	49.4	273	44.0	82,860	376.7
4248	51.4	561	46.6	86,460	377.2
9600	54.5	1505	50.3		
33,120	59.2	4500	54.3		
43,020	60.1	10,380	58.0		
84,300	62.8	31,980	62.6		
86,340	63.1	69,900	66.2		
		85,260	66.9		
		86,520	66.8		

TABLE A (Continued)

TIME-DEFORMATION DATA IN CREEP AND RECOVERY TESTS

TEST 45 (continued)		4530	169.6	201	82.7
		7140	176.4	268	85.0
		10,500	182.4	665	92.0
First-recovery		12,900	186.0	1232	99.1
		15,960	189.0	2010	103.8
Seconds	Mils	29,040	200.1	3116	108.4
		39,060	205.8	4660	113.1
2348	94.9	75,720	217.6	7080	118.3
3432	97.3	102,200	220.0	10,140	122.2
4772	99.5	126,600	225.9	23,340	134.2
6362	101.1	162,500	229.5	33,240	139.3
11,760	104.2	203,100	233.0	69,840	150.3
20,700	107.4	258,900	237.1	96,600	154.1
35,220	110.4			120,900	157.4
45,160	111.7	First-recovery		156,700	160.3
86,220	115.3			197,500	163.3
		Seconds	Mils	259,500	166.4
TEST 46					
		106	54.4	First-recovery	
Specimen 47-11		247	56.8		
Load, 3500		732	60.7	Seconds	Mils
Stress, 3.39		2108	64.7		
R.H., 50% a.d.		3142	66.2	96	46.0
300 g.d., 4.4 mils		7560	70.2	254	48.4
		12,000	72.0	1357	52.9
First-creep		17,220	73.9	4110	56.6
		38,040	77.5	5820	57.8
Seconds	Mils	75,900	79.9	11,040	60.4
		100,200	81.7	31,860	64.2
18	97.6	259,100	84.2	69,840	66.8
35	105.6			94,020	68.4
55	110.5			175,400	71.2
75	113.9	TEST 47		259,600	72.2
93	116.8				
134	121.0	Specimen 48-10			
155	122.7	Load, 3500			
185	124.6	Stress, 3.45			
247	128.3	R.H., 50% a.d.			
318	131.7	300 g.d., 3.3 mils			
404	134.6	First-creep			
555	138.8				
702	141.9	Seconds	Mils		
867	144.8				
1135	148.3	27	68.5		
1485	152.6	53	72.7		
1972	156.4	91	76.7		
2610	160.8	145	80.3		

TABLE A (Continued)

TIME-DEFORMATION DATA IN CREEP AND RECOVERY TESTS

TEST 48		TEST 49		TEST 50	
Specimen 51-2		Specimen 52-12		Specimen 47-8	
Load, 3500		Load, 3500		Load, 3500	
Stress, 3.41		Stress, 3.44		Stress, 3.39	
R.H., 50% a.d.		R.H., 50% a.d.		R.H., 50% a.d.	
300 g.d., 3.1 mils		300 g.d., 3.0 mils		300 g.d., 4.4 mils	
First-creeep		First-creeep		First-creeep	
Seconds	Mils	Seconds	Mils	Seconds	Mils
12	48.1	15	57.8	18	97.8
45	53.2	42	63.1	35	103.9
63	54.6	68	64.6	50	107.4
86	56.1	136	69.4	72	111.3
151	58.9	220	72.5	94	114.0
237	61.1	415	76.6	148	119.0
412	64.2	1017	83.4	220	123.8
1204	72.3	1652	87.0	266	126.1
1972	76.9	4540	95.9	331	128.8
4910	84.2	7020	100.4	395	130.9
7730	89.1	10,860	104.5	1000	144.7
21,580	99.6	18,660	109.8	1780	157.6
28,120	105.2	30,420	115.0	3750	164.5
64,660	115.9	39,180	117.8	5410	170.0
91,560	119.6	78,120	125.6	8340	177.5
115,900	122.1	86,340	126.7	15,900	188.2
151,500	126.3			22,860	194.5
192,400	129.1			86,340	217.1
259,200	132.5				
First-recovery		First-recovery		First-recovery	
Seconds	Mils	Seconds	Mils	Seconds	Mils
		105	44.1		
		306	47.0		
		570	48.6		
		1200	51.1	105	60.0
		5640	55.5	258	63.7
172	42.3	22,500	60.1	505	66.4
390	44.6	38,040	61.3	846	68.5
910	46.2	78,360	63.3	2165	72.5
1860	47.8	86,340	63.7	7020	77.6
3180	49.9			18,180	81.7
6120	51.8			86,280	87.8
27,000	56.7				
65,160	60.3				
89,160	62.1				
170,600	64.3				
259,200	64.7				

TABLE A (Continued)

TIME-DEFORMATION DATA IN CREEP AND RECOVERY TESTS

TEST 51		TEST 52		TEST 53	
Specimen 28-1		Specimen 30-3		Specimen 31-8	
Load, 2000		Load, 3500		Load, 2800	
Stress, 2.09		Stress, 3.68		Stress, 2.91	
R.H., 63%		R.H., 63%		R.H., 63%	
Reached by ads.		Reached by ads.		Reached by ads.	
from 12% R.H.		from 12% R.H.		from 12% R.H.	
300 g.d., 3.5 mils		300 g.d., 3.5 mils		300 g.d., 3.5 mils	
First-creeep		First-creeep		First-creeep	
Seconds	Mils	Seconds	Mils	Seconds	Mils
17	26.9	17	71.5	17	44.2
37	27.9	39	77.9	42	46.9
74	28.9	58	81.6	62	48.2
104	29.8	85	85.1	104	50.3
165	30.7	156	91.2	256	55.0
380	32.2	213	94.8	646	60.9
917	34.7	299	98.9	832	62.3
2485	38.0	455	103.8	1875	66.4
3855	39.8	917	112.6	9540	84.3
6420	41.6	1655	119.8	18,540	92.2
8880	43.0	2895	126.8	36,300	99.9
16,680	47.0	4225	131.6	81,960	109.4
25,320	49.7	6750	137.5	86,340	109.8
43,080	53.6	14,400	149.0		
86,400	58.6	23,280	156.8		
		41,040	165.8		
		86,340	176.6		
First-recovery		First-recovery		First-recovery	
Seconds	Mils	Seconds	Mils	Seconds	Mils
108	26.9	102	51.2	106	39.7
260	28.0	248	54.3	369	42.5
600	29.1	495	57.2	720	44.1
1063	30.2	990	59.8	1084	45.4
1956	31.2	1958	61.7	2735	48.2
4005	33.2	3120	63.7	33,720	56.3
6420	34.4	7475	67.5	86,340	60.2
9516	35.0	38,460	74.5		
40,500	37.2	86,340	78.2		
86,340	39.1				

TABLE A (Continued)

TIME-DEFORMATION DATA IN CREEP AND RECOVERY TESTS

TEST 54		TEST 55		TEST 56	
Specimen 32-9		Specimen 33-4		Specimen 32-11	
Load, 2800		Load, 4500		Load, 3500	
Stress, 2.95		Stress, 4.67		Stress, 3.68	
R.H., 63%		R.H., 63%		R.H., 63%	
Reached by ads.		Reached by ads.		Reached by ads.	
from 12% R.H.		from 12% R.H.		from 12% R.H.	
300 g.d., 3.5 mils		300 g.d., 3.5 mils		300 g.d., 3.5 mils	
First-creeep		First-creeep		First-creeep	
Seconds	Mils	Seconds	Mils	Seconds	Mils
22	46.7	20	150.5	13	70.6
46	49.8	36	163.0	23	78.2
75	51.9	66	174.3	63	83.9
142	55.0	87	179.8	120	90.6
216	57.6	126	187.5	255	99.7
318	60.0	154	192.2	383	104.2
520	62.9	234	200.5	634	109.8
696	64.8	301	206.1	1490	120.8
1098	67.7	573	219.1	3447	132.0
2365	73.4	790	224.6	10,680	149.7
3070	75.2	1421	235.5	20,040	159.9
4195	77.8	1943	240.8	37,740	171.2
8220	83.9	2793	246.4	86,280	182.6
14,850	89.8	4620	254.6		
21,840	93.9	12,000	272.2	First-recovery	
40,380	101.5	21,180	283.6	Seconds	Mils
86,340	112.1	38,940	295.5	102	51.3
First-recovery		86,340	309.5	257	54.5
Seconds	Mils	First-recovery		688	58.6
Seconds	Mils	Seconds	Mils	1205	61.1
98	40.2	113	69.2	2190	63.3
273	42.7	212	71.9	4200	66.3
1092	47.3	353	74.9	34,160	76.1
1650	48.7	825	79.4	86,220	79.5
3030	50.7	1295	82.3		
4365	51.8	2020	84.7		
10,680	54.7	3537	88.2		
20,700	56.7	5400	90.4		
41,580	59.6	36,360	100.0		
86,340	61.8	86,400	104.8		

TABLE A (Continued)

TIME-DEFORMATION DATA IN CREEP AND RECOVERY TESTS

TEST 58		TEST 59		TEST 60	
Specimen 47-9		Specimen 47-6		Specimen 48-4	
Load, 2800		Load, 2000		Load, 2800	
Stress, 2.72		Stress, 1.95		Stress, 2.73	
R.H., 50% a.d.		R.H., 50% a.d.		R.H., 50% a.d.	
300 g.d., 4.4 mils		300 g.d., 4.4 mils		300 g.d., 3.3 mils	
First-creeep		First-creeep		First-creeep	
Seconds	Mils	Seconds	Mils	Seconds	Mils
10	54.2	11	33.8	19	41.1
22	57.4	25	34.9	33	42.2
38	59.6	51	36.0	60	43.1
53	60.9	85	36.9	131	44.9
111	64.4	223	39.0	217	46.1
248	68.9	778	42.4	429	48.2
662	76.2	1758	44.8	735	50.1
1658	82.8	4180	48.0	1310	52.8
2540	86.0	6325	49.7	3110	56.0
3180	88.4	10,200	51.6	9420	62.2
5470	93.6	23,060	55.7	19,440	66.9
7673	96.7	83,840	63.3	40,320	72.0
11,400	100.2	86,360	63.7	86,340	77.8
24,623	107.9				
86,493	122.7				
First-recovery		First-recovery		First-recovery	
Seconds	Mils	Seconds	Mils	Seconds	Mils
102	45.6	105	31.3	105	37.2
342	48.6	257	32.7	265	39.1
855	51.2	2230	36.6	540	40.3
1713	53.3	4110	37.8	1055	41.7
3540	55.9	6745	38.9	3395	44.5
5415	57.2	8340	39.2	6420	46.2
7110	58.0	16,020	40.6	17,460	48.8
9735	59.3	25,020	41.9	40,860	50.1
17,430	61.2	42,720	42.5	86,340	51.8
22,710	62.7	86,340	43.9		
44,130	63.9				
86,610	66.1				

TABLE A (Continued)

TIME-DEFORMATION DATA IN CREEP AND RECOVERY TESTS

TEST 61		TEST 62		TEST 63	
Specimen 48-2		Specimen 50-5		Specimen 50-6	
Load, 3500		Load, 3500		Load, 2800	
Stress, 3.43		Stress, 3.53		Stress, 2.81	
R.H., 50% a.d.		R.H., 50% a.d.		R.H., 50% a.d.	
300 g.d., 3.3 mils		300 g.d., 3.1 mils		300 g.d., 3.1 mils	
First-creep		First-creep		First-creep	
Seconds	Mils	Seconds	Mils	Seconds	Mils
16	64.6	14	50.3	15	37.7
32	68.8	30	53.2	33	38.8
57	71.6	50	55.3	60	39.7
76	73.0	97	58.1	115	40.7
137	76.5	157	60.2	243	42.1
244	80.3	287	63.3	402	43.6
438	85.0	578	67.6	1506	47.3
685	88.7	1075	71.6	3247	50.5
1135	92.9	2335	87.2	6600	54.0
1710	96.5	4108	82.0	20,460	61.4
2253	98.3	7260	87.3	41,280	66.4
3782	103.2	21,240	98.9	86,370	72.9
5525	107.5	41,940	107.2	First-recovery	
9240	112.9	86,340	116.1		
17,700	120.7	First-recovery			
27,000	124.9				
46,860	131.9				
84,360	139.9				
First-recovery					
Seconds	Mils	Seconds	Mils	Seconds	Mils
97	49.6	100	45.2	95	35.8
286	53.2	660	50.1	795	39.4
910	57.4	1600	53.0	2550	42.7
2225	60.4	3210	55.9	31,200	47.6
4020	62.5	31,860	62.1	42,060	47.8
6540	64.2	42,840	62.5	86,520	49.1
8880	65.1	86,340	64.5		
14,310	66.7				
37,860	69.6				
86,340	71.7				

TABLE A (Continued)

TIME-DEFORMATION DATA IN CREEP AND RECOVERY TESTS

TEST 64		2145	42.5	First-recovery	
		3295	44.0		
Specimen 50-7		5580	45.5	Seconds	Mils
Load, 4500		11,700	48.3		
Stress, 4.53		26,820	52.5	110	48.0
R.H., 50% a.d.		42,720	54.8	422	51.7
300 g.d., 3.1 mils		86,360	58.5	1672	56.3
				2550	57.2
First-creeep		First-recovery		35,400	66.0
				86,340	67.8
Seconds	Mils	Seconds	Mils	TEST 67	
11	108.8	130	31.3	Specimen 48-5	
24	117.4	428	32.7	Load, 3500	
51	126.1	2120	35.6	Stress, 3.43	
76	130.9	3320	36.1	R.H., 50% a.d.	
117	135.8	36,180	40.4	300 g.d., 3.3 mils	
151	138.7	86,400	41.4		
221	143.3	TEST 66		First-creeep	
352	149.1	Specimen 47-4			
482	153.6	Load, 2800		Seconds	Mils
778	160.4	Stress, 2.73		23	63.2
1135	166.1	R.H., 50% a.d.		51	67.3
2540	179.3	300 g.d., 4.4 mils		95	70.8
6040	193.9	First-creeep		154	73.7
- -	rupture			195	75.0
TEST 65		Seconds		442	80.8
		Mils		915	86.6
Specimen 52-8				1610	91.0
Load, 2800				2280	94.0
Stress, 2.69		21	60.7	3476	97.9
R.H., 50% a.d.		39	63.6	4920	101.2
300 g.d., 3.0 mils		55	64.9	11,640	111.2
		72	66.2	16,140	115.0
First-creeep		98	67.7	24,360	120.0
		191	70.8	87,420	136.4
Seconds	Mils	463	75.8	First-recovery	
		674	78.0		
12	32.2	1420	83.7	Seconds	Mils
33	34.0	2580	88.5		
61	35.0	4680	93.6	122	50.7
162	36.5	10,860	101.3	490	54.1
248	37.3	25,980	110.9	1193	57.5
490	39.0	41,880	115.8		
1050	40.5	86,340	124.7		

TABLE A (Continued)
TIME-DEFORMATION DATA IN CREEP AND RECOVERY TESTS

TEST 67 (continued)		First-recovery		2500	50.7
First-recovery		Seconds		4020	52.0
Seconds		Mils		32,580	57.0
				86,340	59.5
		108	58.8	TEST 70	
		734	66.1	Specimen 52-7	
		1350	68.8	Load, 4500	
2588	59.8	2345	70.9	Stress, 4.27	
4450	61.7	4920	73.9	R.H., 50% a.d.	
8800	63.7	10,280	76.3	300 g.d., 3.0 mils	
19,080	66.2	21,540	80.2	First-creeep	
37,440	68.5	39,180	81.7	Seconds	
86,400	70.7	86,460	85.2	Mils	
TEST 68		TEST 69		15	91.0
Specimen 48-1		Specimen 52-4		44	101.0
Load, 4000		Load, 3500		70	104.9
Stress, 3.92		Stress, 3.30		86	106.5
R.H., 50% a.d.		R.H., 50% a.d.		130	109.9
300 g.d., 3.3 mils		300 g.d., 3.0 mils		188	113.0
First-creeep		First-creeep		290	116.9
Seconds		Seconds		450	120.7
Mils		Mils		672	124.3
20	88.7	20	52.9	1240	130.2
41	96.1	42	55.0	2925	139.4
60	99.3	63	56.4	11,550	156.6
110	105.1	86	57.5	19,080	162.7
145	107.8	132	59.0	86,280	182.2
296	115.5	220	61.1	First-recovery	
551	122.6	520	65.3	Seconds	
827	127.3	906	68.2	Mils	
1526	135.2	1388	70.3	97	53.4
2494	141.6	1842	71.9	280	56.5
3221	144.8	3710	76.1	1770	63.4
4866	149.7	12,300	85.7	3300	66.0
9180	158.8	19,800	89.7	31,860	74.0
20,220	170.8	86,340	104.0	38,040	74.4
37,800	179.5	First-recovery		86,340	77.7
86,340	193.1	Seconds			
		Mils			
		180	43.5		
		395	45.6		
		825	47.4		

TABLE A (Continued)

TIME-DEFORMATION DATA IN CREEP AND RECOVERY TESTS

TEST 71		TEST 72		25,620	48.6
Specimen 28-7		Specimen 29-4		43,200	49.8
Load, 5500		Load, 3500		85,620	50.8
Stress, 5.69		Stress, 3.76		TEST 73	
R.H., 23.5%		R.H., 23.5%		Specimen 30-4	
Reached by ads.		Reached by ads.		Load, 3500	
from 12% R.H.		from 12% R.H.		Stress, 3.68	
300 g.d., 2.9 mils		300 g.d., 2.9 mils		R.H., 23.5%	
First-creek		First-creek		Reached by ads.	
Seconds	Mils	Seconds	Mils	from 12% R.H.	
12	85.9	18	40.4	300 g.d., 2.9 mils	
38	94.4	44	41.9	First-creek	
58	97.9	72	42.7	Seconds	Mils
84	100.9	99	43.5	16	38.3
140	106.0	172	44.6	39	39.7
189	108.9	210	44.8	68	40.8
240	111.0	435	46.6	115	41.6
316	113.4	802	48.1	205	42.6
470	117.9	1480	49.8	530	45.4
612	120.8	2200	50.8	981	46.0
749	122.9	2580	51.6	1560	47.2
1309	129.2	3380	72.6	1955	48.0
1656	132.0	4470	53.5	2655	48.7
3049	138.9	5970	54.9	3620	49.8
5274	146.2	8580	57.3	5780	51.5
8200	152.7	13,980	59.4	10,800	53.4
16,200	162.7	20,100	61.0	18,780	57.4
24,420	168.8	26,160	62.6	25,560	58.8
41,940	175.9	48,540	65.8	34,320	60.8
84,300	185.4	84,660	68.9	81,180	65.1
86,340	185.5	86,340	69.3	86,340	65.2
First-recovery		First-recovery		First-recovery	
Seconds	Mils	Seconds	Mils	Seconds	Mils
110	66.4	106	38.2	180	37.1
385	70.4	425	40.5	940	40.0
1108	74.3	1100	43.4	2195	42.8
2660	77.5	2600	44.4	4065	42.9
3825	79.1	4330	45.4	6420	43.7
7085	81.6	6590	46.1	12,060	45.0
15,780	85.0	9400	46.7	18,120	45.6
86,460	91.5	17,460	47.7		

TABLE A (Continued)

TIME-DEFORMATION DATA IN CREEP AND RECOVERY TESTS

TEST 73 (continued)		First-recovery		First-recovery	
First-recovery		Seconds	Mils	Seconds	Mils
Seconds	Mils	90	28.6	102	49.6
		400	29.8	267	51.7
		2060	31.8	1105	55.8
24,120	46.1	3770	32.6	3040	58.4
46,470	47.0	6040	33.0	4155	59.5
86,340	48.1	8890	33.7	7060	60.7
		16,920	34.4	9560	61.7
		25,110	34.7	40,500	64.8
		42,660	35.6	86,460	67.3
		85,140	36.6		
		86,340	36.7		
TEST 74		TEST 75		TEST 76	
Specimen 31-6		Specimens 33-3		Specimen 32-7	
Load, 2800		Load, 4500		Load, 4500	
Stress, 2.93		Stress, 4.67		Stress, 4.71	
R.H., 23.5%		R.H., 23.5%		R.H., 23.5%	
Reached by ads.		Reached by ads.		Reached by ads.	
from 12% R.H.		from 12% R.H.		from 12% R.H.	
300 g.d., 2.9 mils		300 g.d., 2.9 mils		300 g.d., 2.9 mils	
First-creep		First-creep		First-creep	
Seconds	Mils	Seconds	Mils	Seconds	Mils
17	28.9			28	55.5
40	29.8			56	57.8
92	30.6			80	59.1
130	30.8	28	57.1	116	60.2
720	32.9	50	59.0	175	62.0
1730	34.3	85	61.0	267	63.6
2040	34.7	115	62.2	316	64.3
2820	35.4	250	65.6	548	67.1
3940	35.9	510	68.8	765	69.1
5340	36.8	740	70.2	1550	73.1
7920	38.0	1185	73.0	2780	76.3
13,500	39.2	1740	75.0	4140	78.9
19,590	40.2	2370	76.8	6745	83.0
22,020	41.3	2840	77.9	12,480	87.2
48,000	43.3	4380	80.8	18,540	90.3
84,180	45.4	5650	82.7	24,540	93.2
86,340	45.5	8480	85.8	46,920	98.9
		16,920	91.9	83,160	103.3
		86,460	105.6	86,340	103.6

TABLE A (Continued)

TIME-DEFORMATION DATA IN CREEP AND RECOVERY TESTS

TEST 76 (continued)		First-recovery		TEST 78	
First-recovery		Seconds	Mils	Specimen 55-7	
Seconds	Mils	110	50.9	Load, 3500	
		495	55.8	Stress, 3.49	
112	49.4	1825	60.0	R.H., 50% a.d.	
373	52.3	2955	61.7	300 g.d., 2.7 mils	
990	54.4	8580	65.7	First-creek	
2735	56.7	20,580	67.6	Seconds	Mils
4950	58.4	71,760	72.0		
7800	59.5	86,400	72.2	12	33.8
15,840	61.6	Fourth-creek		34	35.0
24,000	63.2	Stress, 4.48		53	36.8
41,580	65.0	Seconds	Mils	71	36.1
84,060	67.0			152	37.3
86,340	66.9	14	46.9	224	38.0
TEST 77		31	48.7	565	39.8
Specimen 55-10		50	49.6	1640	43.2
Load, 4500		195	52.8	3480	46.0
Stress, 4.48		375	53.9	8220	50.0
R.H., 50% a.d.		1308	57.4	16,380	53.2
300 g.d., 2.7 mils		1758	58.5	25,080	56.0
First-creek		3300	60.0	64,760	62.9
Seconds	Mils	6000	62.0	81,000	64.2
		65,100	68.8	86,340	65.0
		86,340	70.8	First-recovery	
		Fourth-recovery		Seconds	Mils
		Seconds	Mils		
17	52.9			112	35.9
31	55.5	1990	41.0		
51	57.3	7620	43.7		
185	63.5	19,740	45.8		
234	64.9	70,950	48.0		
363	67.7	86,460	48.4		
825	72.9				
1650	77.1				
2720	81.6				
4540	86.7				
9360	94.2				
17,430	101.0				
26,160	106.7				
66,420	118.9				
82,080	121.8				
86,400	123.0				

TABLE A (Continued)

TIME-DEFORMATION DATA IN CREEP AND RECOVERY TESTS

TEST 78 (continued)		68	93.3	486	70.4
		123	100.3	1840	75.0
		153	102.9	4890	77.7
Fourth-creep		202	106.6	63,600	87.0
		272	110.6	86,280	89.0
Stress, 3.49		758	124.6		
		1190	130.4	Fourth-recovery	
Seconds	Mils	1393	132.2		
		2650	142.6	Seconds	Mils
18	33.6	7365	159.8		
32	34.1	15,540	172.3	107	59.5
51	34.7	24,240	180.5	710	66.2
116	35.7	64,500	198.3	2040	69.6
825	39.0	80,160	202.2	5940	73.8
1417	40.4	86,340	202.9	64,620	82.5
3060	41.7			86,340	82.9
5760	42.9	First-recovery			
64,830	47.6	Seconds	Mils		
86,340	49.0				
Fourth-recovery		93	63.0		
		520	70.0		
Seconds	Mils	1160	73.7		
		6780	81.7		
140	35.3	19,020	87.1		
600	37.9	70,260	92.4		
2880	40.8	86,400	92.9		
6720	42.2				
65,580	46.9	Fourth-creep			
86,520	47.0				
		Stress, 3.49			
TEST 79		Seconds	Mils		
Specimen 55-12					
Load, 5500		11	58.5		
Stress, 5.45		29	61.0		
R.H., 50% a.d.		41	62.1		
300 g.d., 2.7 mils		74	64.0		
		140	66.0		
First-creep					
Seconds	Mils				
23	82.9				
46	89.4				

TABLE A (Continued)

TIME-DEFORMATION DATA IN CREEP AND RECOVERY TESTS

TEST 82 (continued)		First-recovery		5770	41.0
First-recovery				12,060	42.9
		Seconds	Mils	23,280	44.4
				86,400	48.2
		98	44.9		
Seconds		225	47.4	TEST 85	
		1365	53.9	Specimen 33-2	
94		5490	59.9	Load, 3500	
275		19,080	64.9	Stress, 3.61	
955		34,560	67.8	R.H., 73.5%	
2250		86,460	71.5	Reached by ads.	
3385		TEST 84		from 12% R.H.	
7400		Specimen 32-5		300 g.d., 4.0 mils	
13,620		Load, 2000		First-creep	
24,870		Stress, 2.10			
86,340		R.H., 73.5%		Seconds	Mils
TEST 83		Reached by ads.		23	110.6
Specimen 31-1		from 12% R.H.		49	120.6
Load, 2800		300 g.d., 4.0 mils		72	126.1
Stress, 2.94		First-creep		98	131.1
R.H., 73.5%				128	136.2
Reached by ads.				198	143.4
from 12% R.H.		Seconds	Mils	242	146.9
300 g.d., 4.0 mils		20	32.9	430	156.3
First-creep		46	34.9	920	169.0
		73	36.3	1394	175.1
Seconds		108	37.4	2960	186.6
		325	40.8	4980	194.0
18		565	42.7	8730	202.3
37		2140	49.3	15,240	212.9
60		4260	53.8	23,640	220.3
89		7890	59.5	86,340	239.0
128		14,400	64.6	First-recovery	
268		22,800	69.2		
410		81,900	82.0	Seconds	Mils
840		86,340	82.1	98	56.3
1853		First-recovery		250	60.9
4200				1300	68.9
10,620		Seconds	Mils	2335	72.2
21,840		70	30.1	6600	77.1
80,100		475	34.2	12,870	80.1
86,380		1600	37.4	24,120	83.7
				86,340	90.4

TABLE A (Continued)

TIME-DEFORMATION DATA IN CREEP AND RECOVERY TESTS

TEST 86		TEST 87		TEST 88	
Specimen 36-5		Specimen 36-3		Specimen 36-7	
Load, 3500		Load, 4500		Load, 5500	
Stress, 3.70		Stress, 4.71		Stress, 5.82	
R.H., $\frac{1}{4}$		R.H., 50% a.d.		R.H., 50% a.d.	
300 g.d., 3.0 mils		300 g.d., 3.0 mils		300 g.d., 3.0 mils	
First-creeep		First-creeep		First-creeep	
Seconds	Mils	Seconds	Mils	Seconds	Mils
10	45.3	18	72.1	10	121.9
28	48.1	28	75.1	27	137.0
45	49.2	45	78.3	49	147.0
70	50.5	58	80.1	68	152.1
118	52.3	83	82.4	90	156.6
165	53.4	120	85.2	115	161.1
226	54.4	150	87.0	142	164.8
362	56.2	220	90.0	171	167.9
650	58.8	313	93.0	300	177.1
1050	60.6	460	96.8	370	180.9
1945	64.1	788	102.9	666	190.9
2640	65.3	1107	106.2	832	194.9
3780	68.2	1620	110.7	1492	205.6
4880	69.8	2965	118.4	2015	211.3
9690	75.2	4020	122.2	3070	218.5
43,680	89.2	8980	133.3	8100	236.0
18,240	80.4	17,400	142.6	11,250	241.6
86,370	97.0	42,840	156.5	16,440	248.3
		86,370	167.6	20,580	252.4
First-recovery		First-recovery		First-recovery	
Seconds	Mils	Seconds	Mils	Seconds	Mils
92	44.7	100	56.8	73	69.9
440	48.6	255	60.3	184	74.2
1090	51.5	690	63.5	435	78.4
2365	53.3	1290	66.3	1160	85.2
3630	55.3	2820	70.4	3120	88.0
5000	55.6	4180	71.3	8760	93.6
10,890	57.6	10,020	75.0	21,000	97.6
22,920	59.5	22,080	77.7	44,880	101.3
46,800	61.4	45,960	76.3	87,360	104.2
86,400	62.8	86,400	82.8		

TABLE A (Continued)

TIME-DEFORMATION DATA IN CREEP AND RECOVERY TESTS

TEST 89		TEST 89-B		TEST 89-C	
TEST 89-A		Specimen 55-8		Specimen 55-8	
Load, 3500		Load, 4500		Load, 5500	
Stress, 3.50		Stress, 4.50		Stress, 5.5	
R.H., 50%		R.H., 50%		R.H. 50%	
Reached by ads.		Reached by ads.		Reached by ads.	
from 97.8% R.H.		from 97.8% R.H.		from 97.8% R.H.	
300 g.d., 3.3. mils		300 g.d., 3.3 mils		300 g.d., 3.3 mils	
First-creeep		First-creeep		First-creeep	
Seconds	Mils	Seconds	Mils	Seconds	Mils
8	45.4	16	77.3	35	129.7
25	49.2	36	81.5	60	137.2
51	51.8	60	85.3	92	143.0
104	55.1	94	89.1	124	146.5
210	59.0	124	91.1	192	152.1
543	64.6	182	94.5	257	156.0
812	66.9	251	97.4	360	160.6
1120	69.1	620	105.4	485	164.9
2220	74.5	1030	110.0	833	172.7
6600	85.0	1817	115.8	1155	177.3
11,760	90.1	2760	120.2	1525	181.9
17,880	93.9	3550	122.8	2640	190.1
30,660	99.4	4310	124.9	7360	205.3
77,460	109.4	7740	131.3	16,860	218.2
86,400	110.4	17,040	139.9	27,720	224.8
		89,580	158.0	39,420	230.2
					Rupture
First-recovery		First-recovery			
Seconds	Mils	Seconds	Mils		
95	40.3	105	55.1		
745	45.2	350	59.1		
1290	47.4	910	62.7		
8280	53.3	4450	66.8		
17,100	55.6	7440	70.2		
30,660	58.2	28,620	78.6		
80,760	61.3	90,840	82.9		
		185,700	85.5		
		276,400	88.0		
		343,800	88.3		

TABLE A (Continued)

TIME-DEFORMATION DATA IN CREEP AND RECOVERY TESTS

TEST 90		TEST 91		TEST 97	
Specimen 55-4		Specimen 36-9		Specimen 29-7	
Load, 3500		Load, 4500		Load, 3500	
Stress, 3.48		Stress, 4.71		Stress, 3.63	
R.H., 50% a.d.		R.H., 50% a.d.		R.H. 83%	
300 g.d., 2.7 mils		300 g.d., 3.0 mils		Reached by des. from 97.6 R.H.	
First-creeep		First-creeep		300 g.d., 6.0 mils	
Seconds	Mils	Seconds	Mils	First-creeep	
19	38.5	13	72.7	Seconds	Mils
29	39.1	32	78.5	19	241.6
41	39.7	56	82.5	43	257.7
68	40.6	75	84.8	72	268.6
111	41.6	110	87.5	93	273.2
275	43.6	285	95.7	107	275.4
688	46.4	450	100.0	122	277.9
1180	48.3	1335	112.8	150	280.2
1849	49.6	2350	119.5	188	283.8
2940	51.6	3165	122.9	215	286.2
4820	53.6	4035	126.0	280	290.4
9150	56.6	7740	134.6	405	296.2
14,430	59.6	13,260	142.0	640	301.7
21,360	62.1	20,040	147.5	860	305.1
38,160	66.2	24,240	150.7	1185	308.9
86,400	72.7	37,080	156.4	1500	312.0
		86,400	169.1	2070	315.4
First-recovery		First-recovery		2550	318.3
Seconds	Mils	Seconds	Mils	3240	321.8
95	37.7	93	57.2	3880	324.1
257	39.7	250	60.3	4870	326.4
1280	43.6	830	65.0	5895	328.6
2170	44.8	2090	68.8	7440	331.2
6630	47.5	4600	73.1	9240	334.2
14,640	49.2	13,440	76.8	10,800	336.2
25,980	50.3	24,600	79.0	14,940	340.5
35,460	51.5	26,940	79.9	20,100	344.4
87,400	52.8	86,640	83.6	24,090	346.9
				46,080	355.0

TABLE A (Continued)

TIME-DEFORMATION DATA IN CREEP AND RECOVERY TESTS

TEST 98		TEST 99		TEST 100	
Specimen 29-8		Specimen 29-6		Specimen 29-5	
Load, 3500		Load, 3500		Load, 3500	
Stress, 3.76		Stress, 3.74		Stress, 3.74	
R.H. 50%		R.H. 73.5%		R.H. 63%	
Reached by des.		Reached by des.		Reached by des.	
from 97% R.H.		from 97% R.H.		from 97% R.H.	
300 g.d., 3.8 mils		300 g.d., 4.5 mils		300 g.d., 4.0 mils	
First-creeep		First-creeep		First-creeep	
Seconds	Mils	Seconds	Mils	Seconds	Mils
15	72.8	13	152.5	13	103.9
40	79.3	52	173.5	23	110.9
61	82.8	83	180.6	42	117.8
93	86.3	154	190.1	70	124.2
141	90.2	180	192.3	133	132.2
253	95.9	205	193.9	196	137.2
2000	118.0	253	196.9	453	148.2
2460	120.1	450	204.8	623	152.0
3215	122.9	770	212.8	1065	159.0
3990	125.3	1100	217.7	2235	168.2
6150	129.9	1735	223.9	4020	176.0
9960	135.4	2810	230.3	6480	182.0
15,600	140.7	4740	237.3	13,200	191.0
27,060	147.3	5670	239.4	20,580	196.9
42,780	154.3	7260	242.5	37,260	203.8
86,400	160.9	9540	245.6	84,900	213.2
First-recovery		First-recovery		First-recovery	
Seconds	Mils	Seconds	Mils	Seconds	Mils
96	48.5	17	57.0	20	50.6
258	51.6	97	65.0	100	55.9
615	54.5	315	71.4	240	59.4
2465	60.2	612	74.9	515	62.6
3740	61.9	1415	79.7	1095	66.2
7600	65.0	2980	94.0		
19,980	68.4				
26,760	69.5				

TABLE A (Continued)

TIME-DEFORMATION DATA IN CREEP AND RECOVERY TESTS

TEST 100 (continued)		2670	62.8	TEST 104	
		4500	64.8	Specimen 42-4	
		8610	67.8	Load, 4500	
First-recovery		65,580	76.3	Stress, 4.90	
		86,580	77.2	R.H., 50% a.d.	
Seconds	Mils	TEST 102		300 g.d., 3.0 mils	
2030	69.0	Specimen 29-9		First-creeep	
6420	75.2	Load, 3500			
15,900	79.6	Stress, 3.76		Seconds	Mils
26,160	82.2	R.H. 23.5%			
85,140	87.4	Reached by des.		11	68.4
TEST 101		from 97% R.H.		26	73.7
Specimen 29-10		300 g.d., 3.7 mils		37	76.0
Load, 3500		First-creeep		55	78.9
Stress, 3.76				90	82.8
R.H. 50%		Seconds		113	84.3
Reached by des.		Mils		245	90.7
from 97% R.H.				515	97.9
300 g.d., 3.8 mils				890	105.8
First-creeep				1462	111.4
				2680	120.8
Seconds	Mils			4410	126.9
				8400	135.9
18	79.0			30,480	154.8
48	87.6			69,300	167.8
85	92.4			90,420	171.5
128	96.4			159,500	179.8
287	104.7			255,400	186.0
550	111.2			335,600	189.6
1270	121.0			346,700	189.8
4800	136.8			346,800	Recovery
8880	143.9			First-recovery	
24,720	160.2				
68,220	173.3			Seconds	Mils
86,340	176.0				
First-recovery		First-recovery		88	51.5
				210	53.8
Seconds	Mils	Seconds		580	56.8
		Mils		1340	60.5
80	50.6			4260	65.0
335	54.4			11,880	69.2
750	58.0			21,600	72.0
1815	61.0			76,140	77.1
				154,400	80.2

TABLE A (Continued)

TIME-DEFORMATION DATA IN CREEP AND RECOVERY TESTS

TEST 104		85,880	105.0	660	94.2
		171,400	105.6	3680	100.7
		260,800	106.0	15,410	104.9
First-recovery		349,500	106.6	44,870	104.3
		453,400	106.8	81,770	105.9
Seconds	Mils	629,100	106.7	167,100	110.2
		880,900	106.6	256,600	110.8
369,400	84.0			345,300	111.5
676,700	85.5	TEST 114		449,200	111.9
1,030,000	86.8	Specimen 42-7		624,900	112.1
1,708,000	88.5	Load, 5500		876,500	112.4
1,971,000	89.0	Stress, 5.95		TEST 115	
TEST 113		R.H. 50% a.d.		Specimen 42-6	
		300 g.d., 3.0 mils		Load, 5500	
Specimen 42-8		First-creep		Stress, 5.95	
Load, 5500				R.H. 50% a.d.	
Stress, 5.95				300 g.d., 3.0 mils	
R.H. 50% a.d.		Seconds	Mils	First-creep	
300 g.d., 3.0 mils					
				Seconds	Mils
First-creep		19	121.3	5	104.0
		42	133.9	30	126.8
		64	139.7	54	136.2
Seconds	Mils	93	145.5	79	142.0
		127	150.9	110	147.1
6	106.2	195	157.4	135	150.3
27	122.6	293	164.0	172	154.1
50	134.7	408	169.1	366	168.0
71	140.2	515	173.5	603	178.5
92	144.2	624	176.8	930	184.1
100	Recovery	810	181.0	1430	191.6
First-recovery		976	184.1	2020	197.1
		1130	186.0	3660	206.7
		1647	192.6	6140	215.6
Seconds	Mils	1965	194.8	10,920	225.1
		1990	Recovery	17,400	233.8
64	92.6	First-recovery		19,680	236.3
242	95.7			20,000	Recovery
350	96.9	Seconds	Mils		
510	97.4				
1820	100.0	55	82.9		
4400	101.5	155	87.3		
7940	102.5	350	91.4		
15,920	103.6	660	94.2		
45,500	104.7				

TABLE A (Continued)

TIME-DEFORMATION DATA IN CREEP AND RECOVERY TESTS

TEST 115 (continued)		1390	80.0	16,560	77.4
		6340	81.0	63,840	80.1
		19,360	81.3	168,100	81.7
First-recovery		38,800	82.0	252,400	82.3
		83,980	82.0	343,800	82.5
Seconds	Mils	172,600	82.7	424,100	82.7
		277,200	83.0		
85	76.2	360,800	82.9	TEST 118	
330	83.2	702,600	82.9	Specimen 42-9	
750	87.0	788,300	82.9	Load, 3500	
1360	90.0			Stress, 3.80	
2270	92.6			R.H., 50% a.d.	
3970	95.0			300 g.d., 3.0 mils	
23,370	102.9	TEST 117		First-creeep	
68,550	107.0	Specimen 42-10			
96,510	108.0	Load, 4500			
157,300	110.0	Stress, 4.87			
261,300	111.3	R.H., 50% a.d.			
345,500	112.1	300 g.d., 3.0 mils		Seconds	Mils
437,000	112.7	First-creeep		7	41.0
518,400	113.8			20	42.6
684,900	113.4	Seconds	Mils	45	44.0
				62	44.7
TEST 116		28	71.2	90	45.5
		65	75.3	188	47.1
Specimen 42-5		110	79.3	300	48.5
Load, 5500		137	80.6	416	49.5
Stress, 5.95		215	84.0	500	50.3
R.H. 50% a.d.		315	87.0	1460	54.1
300 g.d., 3.0 mils		565	91.7	3240	58.1
		725	94.1	8100	63.6
First-creeep		1160	98.1	9960	65.0
		2140	103.6	10,000	Recovery
Seconds	Mils	2880	107.9	First-recovery	
		3735	110.8		
3	96.0	5655	115.6		
5*	103.0*	10,080	123.0	Seconds	Mils
* Estimated		10,100	Recovery		
5 Recovery				85	43.0
First-recovery		First-recovery		370	46.2
				930	48.0
Seconds	Mils	Seconds	Mils	1380	48.9
				14,220	52.0
80	77.0	80	60.4	61,680	53.7
202	78.1	290	64.7	165,700	54.5
523	79.2	950	68.9	250,000	54.1
		2145	71.6	341,500	54.5
		3390	73.3	508,100	54.4

TABLE A (Continued)

TIME-DEFORMATION DATA IN CREEP AND RECOVERY TESTS

[illegible]

TABLE A (Continued)

TIME-DEFORMATION DATA IN CREEP AND RECOVERY TESTS

TEST 129 (Continued)		TEST 132		66,840	41.0
First-creek		Specimen 28-9		89,370	42.1
Seconds	Mils	Load, 800		158,500	43.5
		Stress, 0.82		425,100	45.9
Seconds	Mils	R.H., 94%		1,470,000	47.7
		Reached by ads.		1,627,000	48.6
Seconds	Mils	from 50% R.H.		2,342,000	48.9
		300 g.d., 7 mils		3,178,000	48.2
256	82.9			TEST 133	
385	86.8	First-creek		Specimen 33-9	
735	92.9	Seconds	Mils	Load, 4500	
1200	97.9			Stress, 4.65	
1920	101.7	15	25.6	R.H., 50% a.d.	
2365	105.2	34	27.5	300 g.d., 3.0 mils	
3660	106.5	51	28.2	First-creek	
4740	107.8	78	29.3	Seconds	Mils
6900	109.6	142	30.9		
7440	111.6	216	32.1	20	88.0
8040	113.5	400	33.6	55	96.5
9120	116.3	710	35.5	77	99.6
10,620	119.8	2010	40.3	100	102.0
19,260	135.8	3210	41.8	355	115.1
27,600	143.8	5190	43.5	440	117.1
67,080	156.3	5820	46.0	545	119.2
72,840	157.3	6120	47.0	1230	129.8
86,460	159.3	6570	48.5	1530	132.1
First-recovery		7620	51.3	2740	140.2
Seconds	Mils	9120	55.3	3510	143.7
		17,610	64.6	6330	151.7
85	42.6	26,100	69.8	8160	155.9
500	49.5	66,340	76.6	11,280	160.3
880	53.3	72,340	77.6	18,600	168.0
1820	57.7	86,400	79.5	28,620	174.4
3470	63.0	First-recovery		39,000	179.5
8160	64.7	Seconds	Mils	87,420	193.7
26,340	69.6			145	24.2
68,400	72.7	370	27.1		
90,900	74.2	980	30.0		
159,800	75.5	2385	32.5		
426,400	78.5	6870	35.1		
1,471,000	81.6	24,480	38.1		
1,631,000	82.0				
1,817,000	82.3				
2,321,000	82.6				
3,179,000	82.5				

TABLE A (Continued)

TIME-DEFORMATION DATA IN CREEP AND RECOVERY TESTS

TEST 133 (continued)		First-recovery		First-recovery	
First-recovery		Seconds	Mils	Seconds	Mils
Seconds	Mils				
170	62.4	65	33.4	60	64.4
315	64.7	440	36.8	125	67.2
790	68.7	1140	37.8	770	74.9
2160	73.3	4830	41.1	1560	78.0
6770	78.4	7320	42.1	7050	81.8
9420	79.6	18,360	43.6	14,580	88.2
20,280	82.8	29,940	44.0	26,040	90.9
31,980	84.7	84,900	45.7	81,120	95.2
87,060	88.5	173,100	46.2	126,000	97.0
132,000	89.8	342,800	47.4	339,100	100.5
344,700	92.6	520,300	47.7	516,400	101.8
1,993,000	98.2	1,991,000	48.5	1,987,000	106.0
TEST 134		TEST 135		TEST 136	
Specimen 30-1		Specimen 32-4		Specimen 33-1	
Load, 2800		Load, 5000		Load, 3500	
Stress, 2.96		Stress, 5.21		Stress, 3.62	
R.H., 50% a.d.		R.H., 50% a.d.		R.H., 50% a.d.	
300 g.d., 3.0 mils		300 g.d., 3.0 mils		300 g.d., 3.0 mils	
First-creep		First-creep		First-creep	
Seconds	Mils	Seconds	Mils	Seconds	Mils
14	33.4	19	105.7	13	44.9
32	34.5	41	113.8	31	47.5
56	35.0	67	118.5	57	48.9
110	35.8	90	121.8	58	50.0
190	36.8	137	126.1	181	52.3
340	37.7	185	129.8	900	59.1
538	39.0	347	137.8	1680	62.5
1155	40.6	580	145.0	3460	66.9
3300	43.2	1132	154.0	11,160	74.9
4920	44.8	2410	165.8	20,700	79.8
8160	46.7	4390	175.0	31,140	83.0
15,660	49.6	12,000	190.9	80,940	92.9
25,440	51.8	21,660	200.2	86,400	93.4
35,880	53.5	32,100	206.7		
86,340	59.4	81,180	223.0		
		86,460	224.0		

TABLE A (Continued)

TIME-DEFORMATION DATA IN CREEP AND RECOVERY TESTS

TEST 136
(continued)

First-recovery

Seconds	Mils
64	42.5
200	45.3
675	48.4
2700	52.4
13,770	56.4
25,140	57.5
80,340	60.7
125,100	60.5
338,300	62.5
515,500	62.9
1,986,000	65.1

TABLE V

MOISTURE CONTENTS VERSUS RELATIVE HUMIDITY AT 73°F.
As Grams of Water per 100 Grams of Phosphoric
Anhydride-Dried Sample

Saturated Salt Solution Employed	Relative Humidity, %	Specimen Number						
		22-33	40-45	46-47	48-49	50-51	52-53	54-57
<u>Desorption</u>								
Potassium nitrite	48.6	7.38	7.32	7.62	7.35	7.30	7.38	7.72
Magnesium chloride	32.9	5.85	5.87	5.83	5.59	5.58	5.62	5.89
Lithium chloride	11.1	2.99	3.16	3.15	2.90	2.89	2.99	3.11
<u>Adsorption</u>								
Lithium chloride	11.1	2.62	2.77	2.76	2.50	2.49	2.59	2.54
Magnesium chloride	32.9	4.97	5.08	5.11	4.81	4.78	4.93	4.94
Potassium nitrite	48.6	6.55	6.66	6.69	6.38	6.35	6.50	6.57
Sodium nitrite	64.8	- -	8.75	8.75	8.39	8.40	8.49	8.72
Sodium chloride	75.5	- -	10.68	10.69	10.03	10.28	10.33	10.62
Potassium chromate	86.5	- -	13.96	13.93	13.28	13.42	13.40	13.93
Sodium sulfate	97.8	- -	25.4	25.5	24.6	24.5	23.7	25.7

^a The values are reported equilibrium relative humidities over the corresponding saturated salt solutions at 73°F. The value of 97.8% R.H. for sodium sulfate was reported by Bobb (63); all others were reported by Wink (53).